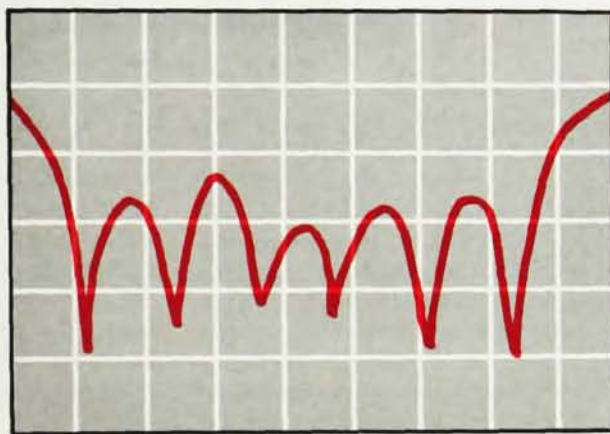


Mössbauer Effect

Recoil-free gamma radiation is used to study relativistic effects, lattice dynamics and hyperfine interactions in solids. Problems encountered in such work are those of low-energy nuclear physics and the need for a precise Doppler modulator.

by Gunther K. Wertheim



TODAY NEW DISCOVERIES in physics are rapidly assimilated. Although only nine years have passed since Rudolf Mössbauer submitted his first article,¹ the Mössbauer effect is currently being used in many undergraduate physics laboratories, and four or five manufacturers are offering off-the-shelf Mössbauer spectrometers.

In the Mössbauer effect nuclei in solids emit low-energy gamma rays without recoiling. The recoil momentum is taken up by the entire crystal. Since the mass of the crystal is so much larger than that of an emitting nucleus, the energy transferred is entirely negligible. So the gamma rays have the full energy of the nuclear transition and widths defined by the lifetime of the decaying state. Further, because the characteristic period of lattice vibrations is much smaller

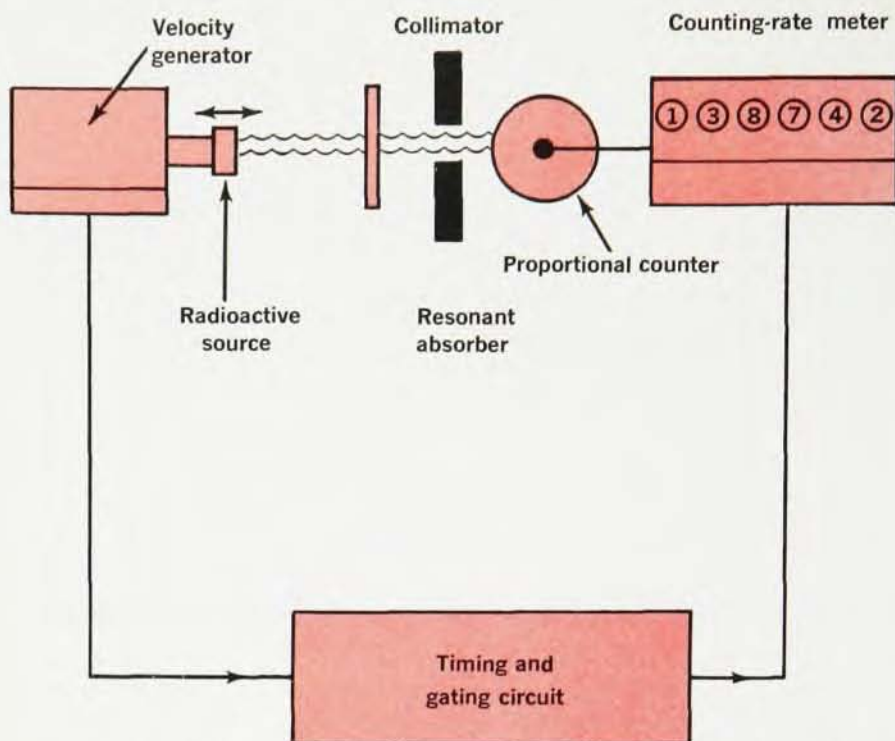
than the nuclear lifetime, such gamma rays are not broadened by thermal motion of the atoms and thus can be resonantly reabsorbed in a similar recoil-free process. This is the essential feature of the Mössbauer effect. (Several introductory books and articles on the effect are available.²)

APPLICATIONS

There is great interest in the Mössbauer effect because the sufficiently small fractional and absolute widths of these gamma rays allow study of relativistic effects, lattice dynamics and hyperfine interactions in solids. Of these applications perhaps the most dramatic was the measurement of the red shift of photons moving in the gravitational field at the surface of the earth where this shift amounts to only 1 part in 10^{16} per meter. Another

application that depends directly on the fractional line width available in the Mössbauer effect is observation of the second-order Doppler effect caused by thermal motion of atoms. This so-called "thermal red shift" has been used in discussion of the twin paradox and provides a direct measure of the mean square *velocity* of atoms in solid. The probability of recoil-free emission or absorption, which is related to the Debye-Waller factor, yields the mean square *amplitude* of thermal motion, and thus provides further information on lattice dynamics.

The most useful applications of the Mössbauer effect depend on the ability to resolve the hyperfine splitting of nuclear ground and excited states. This resolution is possible because hyperfine interaction energies in solids are often greater than the natural line widths of gamma rays originating from the low-lying states that are useful in the Mössbauer effect. The initial interest here is in the nuclear magnetic moments of excited states that are otherwise inaccessible, but the interest does not end when they have been determined. The solid-state physicist and chemist then have a new technique, in many ways complementary to conventional nuclear resonance, with which they can study nuclear hyperfine structure in solids. The tech-



EXPERIMENTAL SETUP typical of Mössbauer-effect studies. —FIG. 1

nique is being applied in the study of magnetism of metals, alloys and compounds, including subjects of current interest such as critical-point phenomena and electron-spin relaxation in solids.

A unique measurement made possible by the Mössbauer effect is that of the change in the nuclear radius between ground and excited states. This parameter is obtained from the shift of the nuclear transition energy with change in chemical environment, generally called the "isomer shift." The isomer shift is the result of the purely electrostatic interaction of the nuclear charge with the s-electron charge density. Widespread chemical application is thus possible since the charge density is related to the details of chemical bonding.

Measurements of the type just men-

tioned are usually carried out in absorption although scattering provides a powerful technique for specific problems.³ A typical experiment (figure 1) requires recoil-free gamma rays that are unsplit by hyperfine interaction and whose energy can be shifted over a small range, the absorber under study and a gamma-ray detector. The output of such an experiment is the counting rate at the detector as a function of the energy shift of the source—that is, an absorption spectrum. The required shifts are only a small fraction of the total energy, no greater than three parts in 10^8 in current experiments, but very high precision is often required. Other problems that occur in such work are those of low-energy nuclear physics such as detection of low-energy gamma rays and data handling. We will only mention them and sketch their solutions briefly.

ENERGY MODULATION

The unique experimental problem that arises in Mössbauer spectroscopy is that of changing the energy of a gamma ray by a small but precisely known amount. The most versatile and convenient techniques are based on the Doppler effect, the principle

used by Rudolf Mössbauer in one of his first experiments.

If an emitter of electromagnetic radiation is approaching an observer with velocity v then the frequency of the radiation ν is increased by an amount $\delta\nu = \nu v/c$, where c is the velocity of light.

The smallest useful velocities are determined by Γ , the natural gamma-ray line width. The ratio $2\Gamma/E$ for Fe^{57} , the most widely used Mössbauer isotope, is 6.6×10^{-13} . This corresponds to a Doppler velocity of 0.02 cm/sec. Measurements can be made to a small fraction of a line width so that velocities of a few microns/sec can be useful, and larger velocities have to be stable and noise free to a similar limit.

Doppler methods

These requirements have given scope to the string-and-sealing wax type of ingenuity that today seldom finds an outlet in physics. This ingenuity is apparent in a few of the devices to be described, but especially in one (figure 2a) in which the velocity is the result of a liquid-level falling as the liquid escapes through a needle valve at approximately constant head.⁴

At first sight it appears impossible to construct a device that will provide a steady energy shift since the source and detector will sooner or later collide, but that is not so. Two devices that have been built provide a steady energy modulation, one based on a tilted rotating disk,⁵ the other on a rotating cylinder,⁶ as shown in figure 2b. In both a component of the tangential velocity provides the Doppler shift, but large-area absorbers are required. These devices are of interest in lab and lecture demonstration.

Far greater convenience is provided by reciprocating motions. Some devices of this type have been based on the slider-crank⁷ or cam-follower^{8,9} combinations familiar to students of machine design. Bearing noise and friction are the major problems, especially in passing through zero velocity where the familiar stick-slip problem enters. These devices produce a complicated velocity wave shape, generally a distorted sinusoidal motion that is of limited utility. The most successful of the early devices were based on the lead-screw mechanism,^{10,11} figure 2c.

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The art of building such devices is of course highly advanced not only for precision machine tools but also for ruling engines. A lathe was in fact used in some experiments.¹² But the Mössbauer effect places a new requirement on these devices; the important parameter is not displacement but velocity, and noise in the velocity is a major problem. Teflon bearings and follower nuts have been instrumental in making these devices successful.

The class of velocity generators that has found most widespread acceptance and is most generally available commercially is based on an electromechanical feedback system.¹³ These devices avoid the sources of difficulty encountered in the purely mechanical ones. They use flexural suspensions and thus avoid the problem of friction. They are velocity rather than displacement controlled so that the desired variable is not the time derivative of the control variable. In addition any desired velocity wave form can be obtained simply by providing the corresponding input voltage wave. The most useful velocity wave forms are constant velocity and linear velocity scan. The latter is generally used in conjunction with a multichannel analyzer that is used to store and add repetitive scans. This most useful form of velocity wave cannot be readily made by purely mechanical means.

These systems can be represented by the simple block diagram, figure 3, of a typical servo system. An electric signal that represents the output velocity is compared with an input signal having the form of the desired velocity wave form. The difference, that is, the error signal, is amplified and used to drive the electromechanical transducer. As every student of feedback and servo systems knows, the problem is to make the system stable, that is, to satisfy Nyquist's criterion and avoid the buildup of spontaneous oscillation.^{14,15} This is accomplished by adjusting the frequency response of the error amplifier. A gain inversely proportional to the frequency is generally a good starting point because the combination of electromechanical transducer and velocity sensor has a transfer function proportional to frequency below resonance.

This relation follows from the dif-

ferential equation for a forced harmonic oscillator (mass on a spring). Below the resonant frequency the dominant force is due to the spring; that is, it is proportional to the displacement. But the output of the velocity sensor is proportional to velocity. (It is well known that the time derivative is represented by a frequency response proportional to the frequency itself.) Above the resonant frequency the dominant force is the inertial force due to the mass, that is, the acceleration. Here the frequency response of the electromechanical system is the reciprocal of frequency. Much of the difficulty encountered in building these Doppler modulators arises from the phase shift that accompanies the change in frequency response at resonance. Systems are usually designed to operate well below the resonant frequency. In general it is desirable to adjust the frequency response of the electronic part of the system to compensate for the peculiarities of the electromechanical one. One modification that has proved successful is to synthesize a driving signal out of components designed to drive the inertia, damping and restoring force terms in the differential equation, and then use the feedback only as a final correction.¹⁶

Regardless of the technique employed, the proper measure of the performance is the ratio of the output velocity voltage to the error signal. In successful systems it has generally been reported to be in excess of 100, adding at most a 1% error to the nonlinearity of the velocity transducer.

A minor problem arises because a velocity-controlled system does not respond to a shift in its neutral position; that is, the feedback transducer is sensitive only to velocity, not to position. In most systems this is taken care of by letting a fairly stiff suspension determine the neutral position. It can be minimized by introducing ac coupling in the feedback amplifier and including the power amplifier within the last feedback loop. In the case of constant-velocity motion this problem can be avoided by operating between limit switches.

The actual components of such a system can be very simple. High-compliance loudspeakers make good electromechanical transducers, especially

when their cones are removed. Loudspeaker components can be recognized even in some commercial Mössbauer velocity modulators. "LVsyn's" (Santborn trademarked velocity sensors) are preferable to loudspeakers as velocity sensors. (The two voice coils found on some loudspeakers cannot be used as electromechanical transducer and velocity sensor respectively because of inductive coupling between them.) Two inexpensive operational amplifiers suffice to construct the error amplifier. An additional current amplifier is needed to match the loudspeaker voice-coil impedance. The pitfalls encountered in constructing such a device are exposed in some of the articles that describe them.

Another type of motion is based on piezoelectric transducers.^{17,18} These are not employed in feedback loops and are generally limited to small velocities unless driven at their resonant frequency.

Calibration

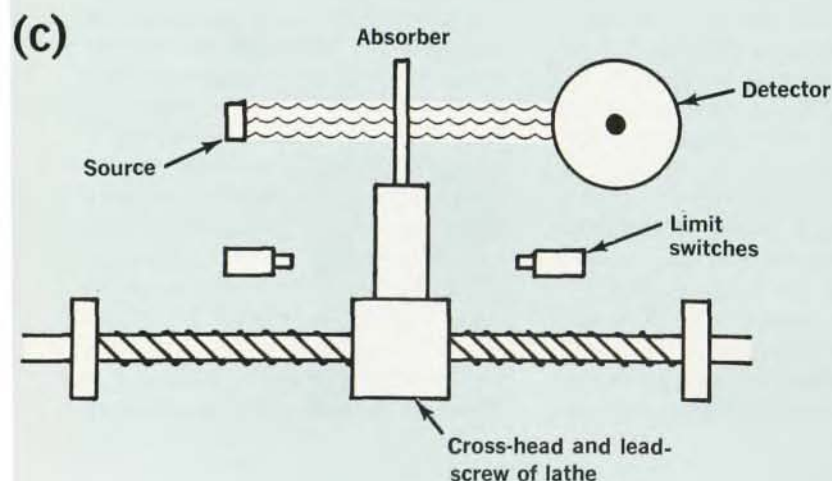
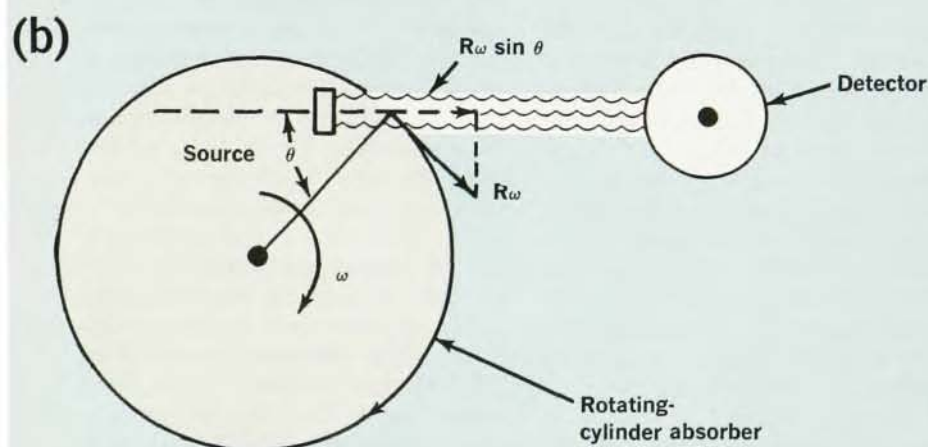
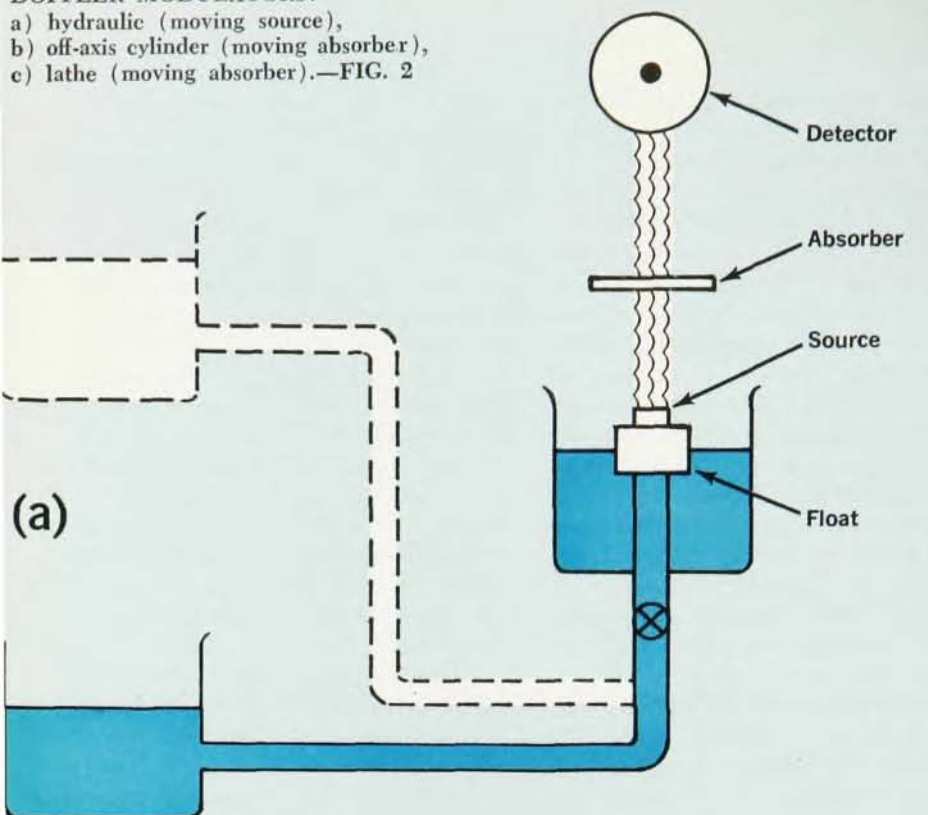
The velocity calibration of these Doppler modulators has been an unexpected challenge to the experimentalist. This fact becomes apparent when it is recognized that most instruments are never checked against the fundamental standards of length and time but are calibrated by running some standard spectrum, perhaps α -Fe₂O₃ or α -Fe. The former, predicated largely on the ready availability of Fe⁵⁷-enriched material, is an unfortunate choice because there are no simple relationships between line positions. The latter is satisfactory provided very pure iron is used. It has only recently been pointed out in print that Fe⁵⁷-enriched iron foils often have reduced splittings and asymmetrically broadened lines.¹⁹

Direct calibration using length and time presents little difficulty in constant-velocity motion with reasonable amplitude, especially if the motion operates between limit switches. It requires considerable ingenuity if the motion is sinusoidal or parabolic and if the constancy of a nominally constant velocity is to be checked to a fraction of a percent. Such checking might require measuring velocity increments down to 1 micron/sec.

The most convenient technique is based on the light-shutter or moiré ef-

DOPPLER MODULATORS:

- a) hydraulic (moving source),
- b) off-axis cylinder (moving absorber),
- c) lathe (moving absorber).—FIG. 2



fect produced by two optical gratings illuminated by a parallel beam of light, that is, without making use of diffraction. If one is fixed and the other attached to the moving element, the transmitted light will be modulated with a frequency given by the velocity divided by the grating spacing. The resulting light pulses can be detected and fed into a time-synchronized multichannel analyzer, thereby producing a direct picture of velocity versus time during the motion.²⁰ Another technique uses interference fringes.²¹

Other methods are less direct and rely on an intermediate calibration step. For example, a magnet-and-coil transducer can be calibrated with a constant-velocity lead-screw device and can then be used to measure more complicated wave forms. Systems based on the capacitance in a resonant circuit have the great disadvantage that they measure displacement rather than velocity. A very simple check of a slow constant-velocity motion can be made by simultaneously observing through a microscope a stationary and a moving grating illuminated by a stroboscope.

SIGNAL-TO-NOISE IMPROVEMENT

The primary information in a Mössbauer experiment resides in gamma-ray quanta whose intensity is related to the Doppler velocity. The next step in data acquisition is to obtain a velocity spectrum: the counting rate or photon flux as a function of photon energy. It was pointed out to me by a spectroscopist, when I was considering the purchase of a multichannel analyzer for this purpose in the early days of the Mössbauer effect, that a simple device has long been used for that purpose in his trade, namely photographic film. Although I was able to counter that argument at the time, it is interesting to note that such a spectrometer has now been built for the Mössbauer effect.²²

Perhaps it should be pointed out that in the present context the *noise* is of statistical origin, arising from the limited number of quanta that are detected. It is not of instrumental origin. The simplest approach is the point-by-point velocity scan in which one accumulates good statistics at one velocity and then progresses to the next. The only difficulty, but one

that is not insuperable, is that it may take many hours to complete a spectrum so that one puts severe requirements on the stability of the gamma-ray detecting equipment. A better approach is to scan quickly but repeatedly, storing and adding up the counts in discrete velocity channels. This requires a multichannel analyzer or digital computer capable of handling counting rates of 5000 events/sec.

Synchronization

The major problem is the synchronization between the storage channels and the velocity. At first sight the most appealing approach is to use the velocity-voltage signal to select the analyzer channel. (Such a system for this widely used technique, and its limitations, has been discussed.²³) Some analyzers provide for input of such a voltage; in others an external pulse-height modulator must be employed. In either case the analyzer is operated in the pulse-height mode. A number of difficulties have made this approach less and less popular. Any phase lag between the physical motion of the source and the electrical velocity signal broadens (or doubles) the lines, and any nonlinearity in the motion or in the analyzer appears as a curvature in the no-absorption spectrum, as does any analyzer channel-width variation. In addition there is an undesirable dead-time effect.

An alternative approach is to let the advance of the analyzer channel be controlled by a crystal clock so that all the channels accept data for the same length of time. The most direct linkage between the motion and the analyzer then is to use the channel address information to control the velocity. There are two common alternatives: One is to use the address voltage itself to produce a velocity sweep. This technique requires a velocity reversal at the end of the sweep that can set up vibrations in the mechanical system. It also results in data having a base-line distortion of geometrical origin. Both problems are avoided if the analyzer is programmed to sweep up and down, as in figure 4. The other is to obtain a symmetrical square wave representing the two halves of memory from the address scaler. It is integrated to produce a symmetrical sawtooth voltage that can be used as in-

put to the electrochemical Doppler modulator. The only drawback here is that the integration of the square wave will introduce some distortion into the symmetrical sawtooth since the integrator must have a low-frequency cutoff. The major drawback common to these two methods is that the motion is operative only while the analyzer is scanning so that some time must be allowed for transients to die out after the analyzer is started.

The third alternative is to use time synchronization. The clock-controlled analyzer sweep is triggered at a fixed point during the independently running Doppler-modulator motion. It turns out to be quite simple to generate a symmetrical sawtooth wave form with good frequency stability²⁴ (1 part in 10^4 per day) that can be used to drive the Doppler modulator. Even in a 1000-channel analyzer this corresponds to a shift of only 0.1 channel per day. That the analyzer and motion run independently during each analyzer sweep does not then adversely affect the data.

A novel system that directly produces the derivative of the Mössbauer spectrum was described some years ago²⁵ and has been recently discussed in greater detail.²⁶ It uses an ac-modulated velocity sweep and lock-in detector in a technique modeled upon that used in nuclear and electron paramagnetic resonance. As described it does not require an analyzer but of course does not provide averaging times longer than the time constant of the lock-in detectors. It should be recognized that there is no fundamental advantage in obtaining a derivative spectrum since the maximal information is obtained when the individual gamma-ray events are counted in the more conventional fashion. A derivative spectrum can always be constructed from the normal spectrum, which is available in digital form.

DISTORTION IN THE DATA

Among the sources of data distortion in Mössbauer work are deviations from linear velocity scan, multichannel-analyzer dead time, source-detector distance changes and vibrations.

Base-line distortion

Mössbauer data are subject to many systematic errors resulting from the

nature of the velocity spectrometer. The fundamental requirement is that the spectrum without absorber be flat, but even this simple requirement is seldom met by real devices.

Devices that code velocity into pulse height and use an analyzer in the pulse-height mode generally exhibit a sloping no-absorption spectrum due to channel-number-dependent dead time in the analyzer cycle. This can be avoided by building into the counting circuit a fixed dead time slightly larger than the largest analyzer dead time. However, this may severely limit the maximum counting rate that can be used. That any deviation from a linear velocity scan results in base-line distortion was mentioned above. These facts argue strongly for using the analyzer in the time mode, avoiding both problems.

But there is another more fundamental problem. Devices in which the source is Doppler modulated will have base-line distortion resulting from change in source-detector distance. It is absent only in constant-velocity spectrometers in which the source sweeps through the same distance for all velocities. Even systems in which the motion is applied to the absorber, however, are not entirely free of this difficulty unless the absorber is sufficiently large and sufficiently uniform to avoid vignetting effects.

Line broadening

Natural line width is the often elusive goal of the experimentalist. We consider here only the spurious width introduced by the spectrometer.

All rapid-scanning spectrometers have an inherent channel-width (slit-width) broadening since the velocity is scanned over a finite range while a channel accepts data. If ± 1 cm/sec is used over 200 channels, the width of each channel is 0.01 cm/sec; this is one half of the natural Mössbauer width of Fe⁵⁷!

More insidious are velocity-dependent mechanisms, like the finite-solid-angle effect. If the detector subtends a cone with half-angle α at the source (assumed to be a point source), Doppler shifts ranging from v to $v \cos \alpha$ will be accepted. (The velocity calibration is also modified by this averaging. The effective average velocity $\langle v \rangle$ is reduced below the applied Dop-

pler velocity v by an amount

$$\delta v = v - \langle v \rangle \approx vD^2/16d^2$$

where D is the diameter of the detector and d the source-detector distance.) The broadening is proportional to the velocity and of the order of the shift, that is, about $vD^2/16d^2$. Hence if the outer line of an Fe^{57} metal spectrum is to be broadened by no more than 0.1 of the natural width, the source collimator distance must be four times the collimator diameter.

Mechanical vibrations of the source and absorber supports are another source of line width. Difficulties often arise from dewars used with gamma rays going perpendicular to their vertical axis. This problem is entirely negligible if the gamma rays propagate along the dewar axis.

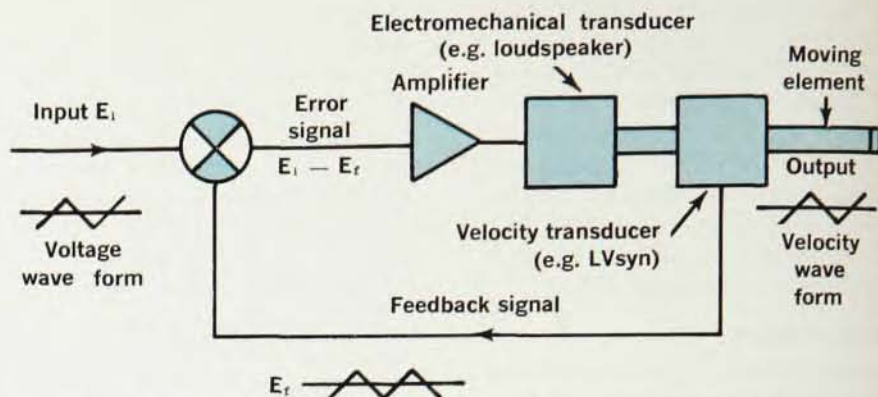
DATA HANDLING

Data handling generally requires computer processing. The conventional form of output, alpha-numeric printed or analog-plotted channel content, is of limited value. One requires output directly compatible with a computer if the computer is not itself used for data taking. Possible modes include punched tapes and cards and magnetic tape. Considerable savings of time and money can be effected if the central computer facility provides magnetic tape storage accessible by data-phone.

Processing of data generally requires such steps as combining halves, correcting for base-line distortion and background, and least-squares fitting. Use of a small on-line digital computer to take the place of the multichannel analyzer has recently been demonstrated.²⁷ Such a machine can be used to perform much of the data processing mentioned and in addition can be used to program the experiment. Least-squares fitting of complicated spectra is not possible on small machines. The economics of using a programmed computer rather than a multichannel analyzer must be critically examined for each case.

GAMMA-RAY DETECTION

The need for a low-noise, high-resolution detection system for gamma rays of energy as low as 6 or 8 keV led to revival of interest in proportional counters. Even with the 14.4-keV



FEEDBACK SYSTEM. Block diagram shows velocity-controlled electro-mechanical feedback system for Doppler modulator. —FIG. 3

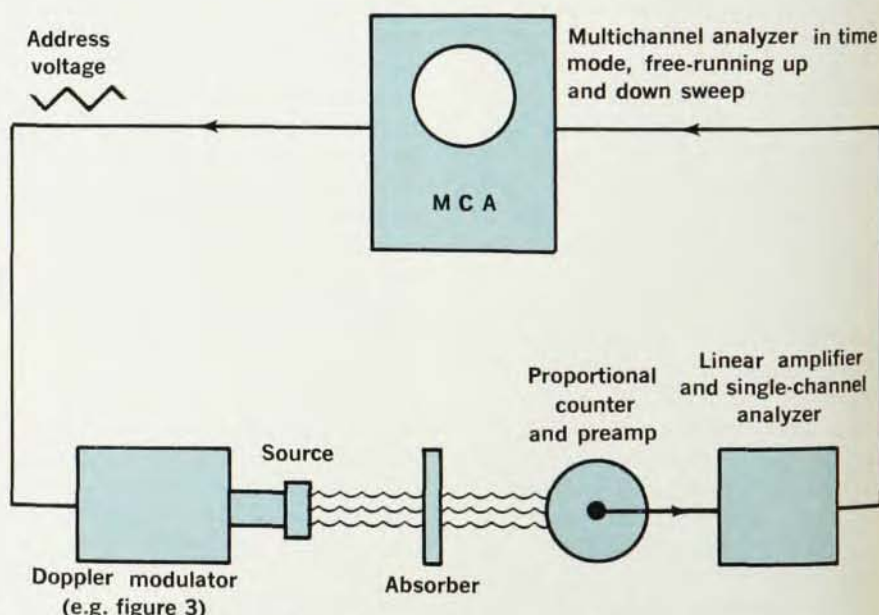
gamma ray of iron for which scintillation counters are satisfactory, the higher resolution of proportional counters is advantageous, especially if x rays from the host crystal fall nearby; for example, a Co^{57} -in-palladium source has a 21-keV x ray that is poorly resolved by most scintillators.

The low resolution of $\text{NaI}(\text{Tl})$ scintillation counters arises from the limited number of photoelectrons produced at the photocathode of the photomultiplier. Efficiency of conversion of incident gamma energy into photoelectrons is low because of the many steps in the energy conversion. The incident gamma ray is stopped by the photoelectric effect in the scintillator. The energy of the electron is converted

into ionization and eventually into excited atomic states. These in turn radiate visible photons, which finally eject photoelectrons from the photocathode.

Proportional counters are inherently superior because the primary ionization in a gas is amplified and collected. The number of ion pairs exceeds the number of photoelectrons by one or two orders of magnitude. Good efficiency for gamma rays of energy up to those of Fe^{57} (14.4 keV) is obtained by using xenon in a 5-cm-diameter counter. The energy range can be extended by resorting to larger counters or filling them to a pressure greater than 1 atmosphere.

Argon-filled counters have found ap-



TIME-MODE SPECTROMETER. Multichannel analyzer is programmed to sweep up and down in controlling velocity. —FIG. 4

plication mainly for work with Tm^{169} and Ta^{181} . They can be used for Fe^{57} if low efficiency or large size can be tolerated. They have the advantage that they stop very few of the higher-energy gamma rays and thus make possible a very simple Mössbauer spectrometer that does not require a single-channel analyzer.

Krypton has its K edge at 14.32 keV, just below the energy of the Fe^{57} gamma ray. It appears that it should be very advantageous in a proportional counter because of its large photoelectric cross section. However the 12.5-keV krypton K x ray is very likely to escape from the counter. Since the fluorescent yield is 61%, only 39% of the incident quanta are likely to be counted. This problem is not serious in a xenon-filled counter since the 4-keV L x ray is readily stopped.

Semiconductors

The greatest promise lies in lithium-drifted germanium and silicon detectors.²⁸ The former have already been used to good advantage to separate closely spaced gamma rays in Mössbauer experiments. The resolution in these devices is limited not by statistics but by the available amplifiers. Resolution somewhat better than 1 keV is currently available with field-effect transistor amplifiers. In actual use these devices are less convenient since they must be operated at liquid-nitrogen temperature, but they are so small that they can be incorporated into the dewar used in the experiment. This incorporation can increase the solid angle and decrease the number of windows that must be traversed.

A unique detection scheme, specific to the Mössbauer effect, has found only limited application. One can build so called "resonant" detectors that use the resonant nuclear cross section of the isotope under study. They consist of a Geiger-counter tube or proportional counter containing a thin foil of the resonant isotope in a chemical form with unsplit nuclear levels. With Fe^{57} , enriched stainless steel is suitable. Detection of a recoil-free gamma ray requires the following events: resonant nuclear absorption, deexcitation of the nuclear excited state by internal conversion, deexcitation of the atomic excited state by emission of a K x ray, which then triggers the Geiger

counter. For Fe^{57} the overall efficiency is quite high since the internal conversion coefficient is about 10 and the fluorescent yield is 35%. The chief advantage is that such a detector is sensitive only to recoil-free gamma rays and thus gives the resonant effect directly.²⁹ Conventional absorption experiments give the effect superposed on a background of nonresonant radiation. A minor advantage is that a single-channel analyzer is not required, making possible very simple demonstration equipment.

Unfortunately such resonant detectors are suitable only for examining the emission spectrum of a source. Even in that case it may well be necessary to apply Doppler modulation to the resonant detector, a not too desirable requirement. If an absorber is to be examined, most of the advantages are lost, unless it is made part of the resonant detector.

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