AN IMPROVED APPARATUS FOR DETECTING PIEZOELECTRICITY

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ABSTRACT

A simple method of detecting piezoelectricity is of value to the mineralogist as an aid in crystal classification.

An instrument is described which is capable of detecting, qualitatively, the pieozelectric effect in crystals ranging in size from approximately 0.1 mm. to 4 cm., having coupling coefficients from approximately 3% up. The apparatus described is simple to operate, and reliable in performance, having given no spurious indications of activity.

The effect of crystal conditions on sensitivity is discussed, and suggestions given for obtaining optimum performance.

Tables are presented showing the size range, and types of materials detected, and the qualitative agreement between the simple "click" indication of this equipment and the more precise measurement on larger oriented specimens.

A complete schematic diagram along with constructional details is given in the appendix.

In the analysis of crystal structure, accessory information concerning the center of symmetry is always of value. Since the presence of a piezoelectric effect is positive proof of the absence of a center of symmetry it was thought an improved apparatus for detecting piezoelectricity would be of interest to mineralogists as an aid in crystal classification. A comprehensive survey of inorganic materials, for example, demonstrated conclusively that an appreciable percentage of the accepted symmetry classifications reported in the literature are incorrect. The device described in this paper is a modification of that developed by Giebe and Scheibe[1] and has proven to be highly reliable in the survey of piezoelectric materials carried out at the Naval Research Laboratory. The apparatus operates on the principle that a piezoelectric crystal placed in alternating electric field will be set into a relatively large vibration whenever the frequency of the field is the same as a natural resonant frequency of the crystal. If the frequency of the field is varied, a momentary vibration of the crystal will be set up as the frequency passes through any of the resonant frequencies of the crystal. This momentary vibration of the crystal reacts with the exciting electric field and can be made to produce a click in headphones or loudspeaker. The results of the measurements with this apparatus were later checked with more accurate measurements of the activity on larger crystals. In all instances checked there was qualitative agreement between the activity as estimated by the vol-

[1] Giebe, E., and Scheibe, A., A simple method for qualitative indication of piezoelectricity of crystals: *Zeist. Phys.*, **33**, 760–766 (1925).

ume of noise produced in this "Giebe-Scheibe" and the more accurate measurements.

This apparatus has never been known to give a spurious indication; that is, if a click is heard it is a definite indication that the crystal is piezoelectric.

In common with other devices of this type, however, it is subject to limitations and under certain conditions may fail to detect piezoelectricity when it does exist. This may be due to one or more of several causes.

(*a*) The crystal fragments may be so small that all resonant frequencies lie above the frequency range of the oscillator.

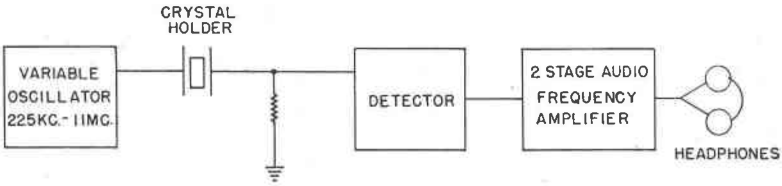


FIG. 1

(*b*) The electrical and mechanical losses in the crystal may be so large that it is impossible to produce sufficient amplitude of vibration to give an indication.

(*c*) The crystal fragments may be of such a shape (e.g. needles or plates) that they are not randomly oriented so that the field is not applied in the proper direction to excite the crystal.

(*d*) The material may be intimately twinned, thus effectively reducing the apparent size to below that which the apparatus can detect. The effects of these limitations may be minimized by observing certain precautions in making the measurements; and by making full use of the known symmetry data, and any other available information concerning the material tested, which might give a clue to which factors could be masking a piezoelectric effect.

DESCRIPTION OF APPARATUS

Figure 1 shows a functional block diagram of the apparatus. The output of the oscillator is placed across the crystal and resistor in series. The voltage across the resistor is rectified and amplified. Any change in voltage across the resistor will have an audio frequency component which will be audible in the headphones. As the slowly changing oscillator frequency approaches and passes a resonant frequency of the crystal, the crystal is set into vibration and undergoes changes in electrical impedance. This causes the voltage across the resistor to vary abruptly which is equivalent to an audio modulation of the oscillator frequency. The vibration of the crystal continues for a short time after the driving frequency has passed the resonant frequency of the crystal, and during this time generates a decaying sinusoidal voltage at its resonant frequency which beats with the exciting frequency. This modulation is then detected and amplified to produce the click in the headphones.

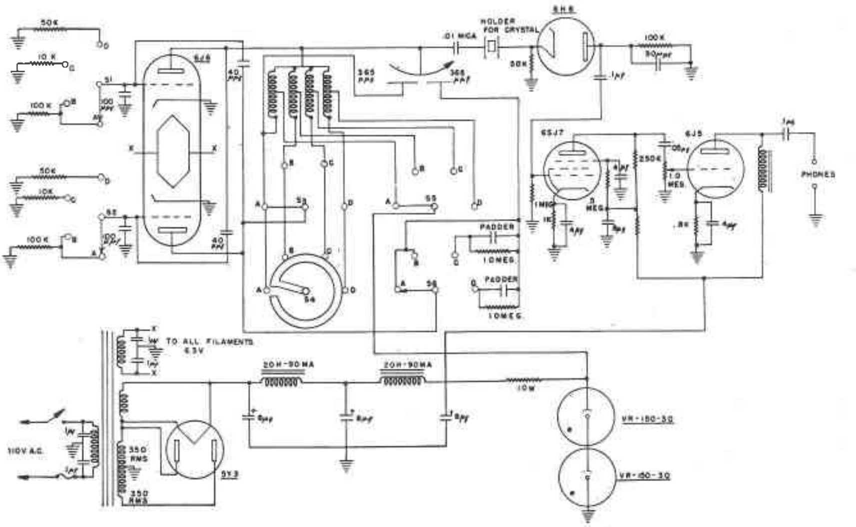
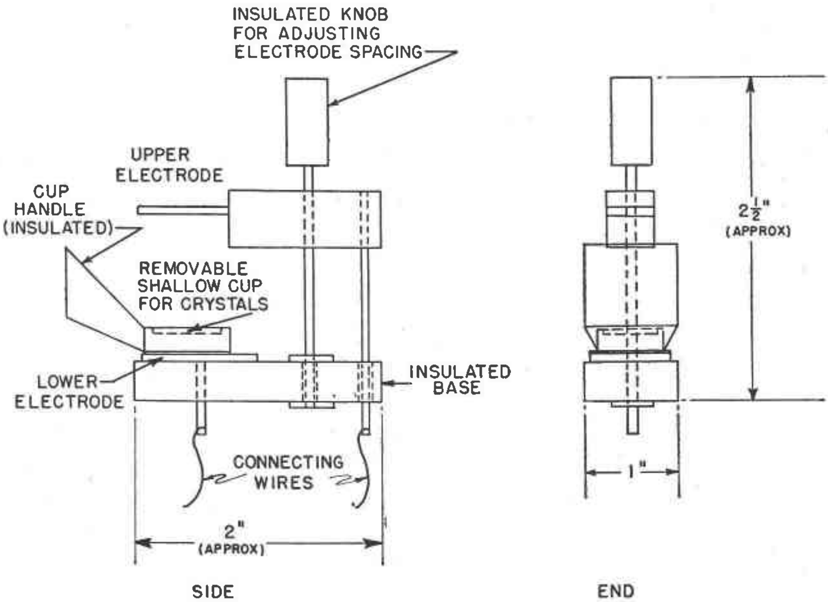


FIG. 2

The amplitude of the click heard in the headphones is determined by the magnitude of the piezoelectric effect; the rate of change of the oscillator frequency; the amount of field across the crystal; the electrical and mechanical losses in the crystal, and the orientation of the crystal. Maximum sensitivity is determined by the signal to noise ratio and is almost constant over the entire range. No clicks are heard when the crystal holder is empty so it is unnecessary to remember any "bad places" on the dial where the response is spurious.

A special metal dish is used to hold the crystals (Fig. 3). This slips between two electrodes of variable spacing which form part of the capacity of the tuned circuit of the oscillator. Variable spacing of the electrodes permits the effective field across the crystals to be varied and facilitates insertion and removal of the dish.

Four frequency bands cover the range 225 kc. to 11 megacycles. Controls for the audio output voltage, oscillator tuning and band switching are on the front panel. The power supply is built into the equipment which is A.C. operated. A complete schematic diagram is shown in Fig. 2. The range was not extended above 11 mc. because the L-C ratio could not be made optimum with the tuning capacitor available, plus the fact that the noise effects increase enormously with frequency above 10 mc. Furthermore, the present range is satisfactory for 100 mesh material in most cases, and tests on smaller particles would be suspect in any event.



SAMPLE HOLDER

FIG. 3

Use of the Apparatus

In using the apparatus small randomly oriented crystal fragments are placed in the metal holder which is then inserted between the electrodes. No cutting, grinding or electroding of the fragments is necessary as in more refined piezoelectric measurements. If possible tiny and imperfect grains should be avoided. The tuning dial is then turned moderately rapidly until clicks are heard in the headphones. Care should be taken to first use a wide spacing in the crystal holder plates so the field across the crystal will be low. This avoids possible breaking of the crystals. If they are broken by too great an excitation it is not possible to reproduce the click at the same point on the tuning dial because the fracture may result in very small fragments and in extreme cases the response may be lost entirely.

If the precautions in construction discussed in the appendix are observed there should be no "spurious" responses; that is, any click heard is a sign of piezoelectric activity in the crystal and is not due to sudden variation of the oscillator or to other extraneous causes.

If no click is heard over the entire frequency range, factors which might prevent detection of the piezoelectric effect should be considered:

(*a*) *Size of the fragments.*—The crystal fragments may be too small. The smallest size detectable will depend on the maximum frequency of the oscillator and the frequency constants of the crystal. In general dense, flexible materials may be detected in smaller sizes than light stiff materials. A list of different kinds of crystals which showed piezoelectric activity with their sizes is shown in Table 1.

TABLE 1. TYPICAL SIZES ON WHICH RELIABLE CLICK TESTS HAVE BEEN OBTAINED

Material	Approximate Linear Dimensions
Ammonium dihydrogen phosphate	0.1 mm. (100 mesh)
Quartz	0.2 mm.
Twinned quartz	1.5 mm.×1.5 mm.×0.5 mm.
Hemimorphite	1 mm.×0.5 mm.×0.1 mm.
Iodic acid	0.15 mm.
Pentaerythritol	0.2 mm.
Potassium dihydrogen phosphate	0.2 mm.
Benzil	0.1–0.2 mm. (100 mesh)
Ammonium oxalate	0.1–0.2 mm. (100 mesh)

It should be mentioned that the fragments may be cracked internally so that the effective size is much less than would be estimated.

(*b*) *Losses in the Crystals.*—Either mechanical losses due to internal friction or electrical losses due to high conductivity might be sufficient to mask a piezoelectric response. Since most crystals have resistivities greater than 10^7/ohm-cm., electrical conductivities high enough to be troublesome would not be expected in most crystals unless a conducting layer is formed on the surface by adsorbed moisture.

Presence of a conducting layer due to moisture ordinarily can be detected by a characteristic cracking and popping noise, and in many cases the difficulty can be eliminated by testing in a dry atmosphere.

High conductivities would be expected to be a factor only in semiconducting materials such as some of the oxides and sulfides. The rectification occurring in the semiconducting materials does not in itself produce a click in this apparatus so that there is no more chance of spurious indications than with other crystals.

Mechanical losses sufficient to affect the test are very rare, and would be suspected only in the case of very soft high molecular weight organic materials.

(*c*) *Improper Orientation.*—The crystal fragments should be randomly oriented. If the original shape is such as to prevent this the fragments should be further broken to permit extensive reorientation.

TABLE 2

Crystal	Crystal Class	Estimated Activity "Giebe-Scheibe"	Piezoelectric Coupling in Per Cent
CuCl	T_d	Active	12*
$NaClO_3$	T	Weak	3
$NaBrO_3$	T	Weak	4
KH_2PO_4	V_d	Moderate	11
$NH_4H_2AsO_4$	V_d	Moderate	24
$NH_4H_2PO_4$	V_d	Strong	30
$NiSO_4 \cdot 6H_2O$	D_4	Weak	6
$NaH_2PO_4 \cdot H_2O$	V	Weak	5
$MgSO_4 \cdot 7H_2O$	V	Weak	3
$ZnSO_4 \cdot 7H_2O$	V	Weak	6
HIO_3	V	Strong	30
$KLiSO_4$	C_6	Slight	5
$LiNaSO_4$	C_{3v}	Slight	8
$KBrO_3$	C_{3v}	Strong	25*
$MgSO_3 \cdot 6H_2O$	C_3	Weak	6
$Li_2SO_4 \cdot H_2O$	C_2	Strong	35

The piezoelectric coupling factor is a quantitative measure of the piezoelectric activity. The value given in the table is the maximum value for the crystal.

* Indicates an approximate value from measurements on small cubes.

(*d*) *Twinning.*—Intimate twinning on a small scale may also mask the piezoelectric effects. Twinned quartz plates, however, which showed no activity on a standard U. S. Army activity meter were easily detected with this apparatus.

Assuming proper attention to technique, the magnitude of the response is reasonably indicative of the piezoelectric sensitivity, because the other properties of the crystal such as mechanical and electrical losses which affect the results are relatively constant in most materials so that the piezoelectric coupling is the factor controlling the amount of response. Table 2 shows a comparison between the response on the small crystal test compared with the results of more refined measurements.

Appendix

Constructional Details for the Giebe-Scheibe Equipment

Figure 2 shows the complete schematic diagram of the equipment. A 10″×14″×3″ chassis is ample for suitable arrangement of all components. The oscillator and detector components should be placed in a well-shielded compartment. The holder for crystal grains may be placed outside of the shielded compartment. Figure 3 shows a simple crystal holder that is not too difficult to construct. The only front panel controls are the tuning dial, the gain control, the band switch, and the on-off switch.

The oscillator is of the push-pull type (6J6) selected because of its ease of adjustment, and the simple type of tank coil used. The feedback is obtained by means of the 40 *mmf* capacitors connected from the plate of one tube to the grid of the other tube. Switch (*S*) changes bands. ($S_{1,2,3,4,4,6}$ are ganged.) The purpose of the switch sections are as follows:

—S_1 and S_2 change grid resistors for class (*C*) bias.
—S_3 and S_5 select coils, *A*, *B*, *C* or *D*.
—S_4 shorts out the unused coils.
—S_6 selects tuning capacitor padders.
Band *A* covers the range from 225 KC to 875 KC.
Band *B* covers the range from 875 KC to 2.8 mc.
Band *C* covers the range from 2.75 mc. to 5.8 mc.
Band *D* covers the range from 5.65 mc. to 11 mc.

If mica padding capacitors are used, shunting them with a (1) megohm resistor will reduce dielectric noise. Both sides of the main tuning capacitor are above ground potential; and therefore must be well insulated from chassis and panel. The A.C. voltage across one-half of the tank coil is 150 v. to 200 v. RMS. This is the voltage across the crystal holder. The amount of field across the crystals may be varied by changing the spacing of the crystal holder electrodes. An inverted (6H6) detector is used in order to reduce the effect of the heater-cathode potential on hum output.

Nothing need be said about the audio frequency amplifier and power supply except that they should be carefully arranged and wired so as to eliminate as much noise and hum as possible. It is not recommended that a speaker be mounted in the cabinet because of acoustic feedback effects.

The ultimate sensitivity of the equipment is determined almost exclusively by the noise level. Attention to the following details is necessary in order to minimize noise and to entirely eliminate spurious "clicks" which may be mistaken for crystal activity.

1. The oscillator section and detector elements must be fully shielded

in order to eliminate beat notes with external signals. Beats sound almost the same as clicks, and therefore must be avoided.

2. Oscillator coils must be shielded, and the unused coils shorted out. If this is not done, the natural resonant frequency of the unusued coils will absorb energy from the used coil and will also cause "broad" clicks.

3. The tuning condenser rotor should be well grounded to the condenser frame with a short lead.

4. Oscillator components must be adjusted so that no sudden changes in amplitude occur when the frequency is varied. Sudden changes in oscillator amplitude will also cause clicks.

5. The oscillator tube should be shock mounted to avoid microphonic effects.

6. Sometimes simply shorting the coils is not sufficient, for it only succeeds in reducing the "Q". If shorting is not sufficient a capacitor may be shunted across the coil instead, to place the natural resonance out of the range being used.