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New Equipment and Procedures for the Maintenance and Comparison of Standards of Electromotive Force at the BIPM[+]

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Abstract

This article describes improved potentiometers, temperature-controlled enclosures and measurement techniques used to relate reference groups of high-quality standard cells to the BIPM (Bureau International des Poids et Mesures) Josephson-effect standard with an uncertainty of less than 10 nV. This performance is demonstrated by the results over the last four years. The same techniques are now being applied in the routine comparisons of international standards of electromotive force by the transfer of standard cells. An analysis of the behavior of the cells that have participated in comparisons over the last 12 years indicates that the uncertainty due to the instability of cells as transfer standards is about 0.5 μV, i.e., one hundred times greater than the best reproductibility in the laboratory. A comprehensive tabulation of uncertainties shows this to be by far the greatest single contribution.

Introduction

Although the Josephson effect has replaced the standard cell as the primary reference standard of electromotive force (emf), in practice less than ten laboratories in the world realize the Josephson-effect standard on a regular basis. Even in these laboratories standard cells are used to maintain the emf standard between Josephson-effect measurements and to make emf comparisons with other laboratories.

The purpose of this article is to describe the equipment and procedures used at the BIPM (Bureau International des Poids et Mesures) to maintain reference standard cells having emf's which are known, with respect to the BIPM Josephson-effect standard, with an uncertainty* of less than 10 nV. Since the beginning of 1981 a new standard cell comparison facility has allowed us to apply these same techniques to the comparison of cells sent to the BIPM from national standards laboratories. As we shall see, the uncertainty in the international comparison of emf standards by the transfer of standard cells is much greater than 10 nV. We describe some of the inadequacies of the equipment used in comparisons and offer some suggestions for improvement. We draw particular attention to the most important and least controlable limiting factor in the use of standard cells for the comparison of emf standards, namely, the large changes which take place in such cells as a result of transport. We conclude with a table of uncertainties associated with such comparisons.

[+]A condensed version of this paper will be given at the Conference on Precision Electromagnetic Measurements in June 1982.

*Throughout this article all uncertaincies are expressed as one standard deviation estimates.

The BIPM Josephson-Effect Standard of emf

The equipment and operation of the BIPM Josephson-effect standard of emf has been described elsewhere [1] but for convenience we include a brief summary here. A Josephson potential of about 10 mV, corresponding to one hundredth of the emf of a standard cell, is obtained by irradiating at 9 GHz two lead-lead oxide - lead tunnel junctions connected in series. This potential is compared with that of a standard cell by means of a double series-parallel comparator containing two sets of ten 100 Ω main resistors closely matched in temperature coefficient as well as in value. A current of 1 mA from a built-in regulated supply produces a potential difference across the 1 000 Ω network of series-connected resistors equal to the standard cell emf. The same current produces a 10 mV potential drop across the 10 Ω resistance string. This potential difference is compared with the junction output. Coarse adjustment of the junction output potential is made by varying the step number of either junction and fine adjustment is obtained by small frequency changes. The detectors in both the standard cell and junction circuits are photocell galvanometer - amplifiers having outputs photocoupled to a pen recorder. Typically, the deflections are integrated over about three minutes. To eliminate small differences between the two resistor sets they are interchanged during the measurements. Thermal emf's and their linear drifts are eliminated by polarity reversals. Thus, four separate pairs of detector readings yield one determination of the standard cell emf. Using the frequency-to-voltage conversion factor recommended by the Consultative Committee on Electricity [2] the cell emf, V_{sc}, is given by

$$V_{sc} = 100nf/483\ 594,$$

where the factor of 100 is the ratio of the comparator, n is the step number, f the average microwave frequency in GHz and V_{sc} is in terms of $V_{76\text{-BI}}$. The symbol $V_{76\text{-BI}}$ is used to indicate "BIPM volts" obtained by the Josephson effect using the frequency-to-voltage conversion factor of 483 594 GHz/V. The "76" in the subscript recalls that this unit was officially adopted by the BIPM in 1976.

A typical run of Josephson-effect measurements now consists of 5 or 6 determinations of the value of one standard cell. In the past, measurements were often made on two cells, one saturated and one unsaturated. Unsaturated cells were used because of their small temperature coefficients. Once we built our own thermoregulated enclosures their use was gradually discontinued. Figure 1 is a histogram of the standard deviations of the mean of all of the 68 Josephson-effect runs made from 1978 through 1981. The average standard deviation of a single run is 7.3 nV.

The run-to-run reproducibility of the Josephson-effect standard may be seen in Figure 2 which is a plot of the behavior of the mean of four high-quality standard cells in terms of $V_{76\text{-BI}}$ since 1978. Each point on the graph was obtained from the Josephson-effect determination of one

standard cell and the intercomparison of standard cells before and after the Josephson-effect measurements. For the period after June 1979 a linear, unweighted, least-squares fit was made to the data. The average residual uncertainty of an observation is 8.5 nV.

The downward drift of the group mean from April 1977 to about July 1979 was very regular and seems to be due to the establishment of equilibrium at 30 °C after prolonged storage of the cells at 20 °C. If U is the mean of the group after correction for the ultimate linear drift of 0.20 nV/d shown in Figure 2, U_o the corrected group mean just after the temperature change from 20 °C to 30 °C, U_f the final constant value of the corrected group mean, and t the time, then we find,

$$U = U_f + (U_o - U_f)e^{-t/\tau}$$

where $\tau \approx 175$ d and $U_o - U_f = 1.6$ μV. This behavior is not an example of the usual hysteresis effect following a temperature change. The approach to equilibrium is much slower and from above in contrast to a rather rapid approach to equilibrium from below in the usual case of hysteresis. This may be a manifestation of the "negative hysteresis" effect mentioned by Hamer [3].

The behavior of standard cells, no matter how predictable, can only serve as a check on the precision of a Josephson-effect standard. The accuracy of the standard is obtained by estimates of known sources of error and by comparison, in so far as this is possible, with other Josephson-effect standards presumed to have different sources of error.

In Part 1 of Table I we summarize the estimated uncertainty contributions for the measurement in terms of the Josephson-effect standard, $V_{76\text{-BI}}$, of one cell of reference group V. Some of the items in this list, for example uncertainties due to the series-parallel comparator, follow from our earlier estimates [1]. Significant improvements over previous values have been made in the standard cell-related uncertainties and the random uncertainty of the mean. The total uncertainty in the Josephson-effect measurement of one cell is about 23.5 nV. In Part 2 we summarize the additional uncertainty in the comparison of the measured cell with the cells of reference groups V and N. The behavior of four of the cells of the former group is shown in Figure 2. In this table the uncertainties related to cells, their enclosures and their comparison apply only to our own saturated cells in our own enclosures.

Comparisons of Josephson standards of emf have been made by transporting the BIPM apparatus to the Physikalisch-Technische Bundesanstalt (PTB), Braunschweig, F.R.G. in April 1975 and to the National Physical Laboratory (NPL), Teddington, U.K. in April 1978. Differences between Josephson-effect determinations of standard cells by the two systems were (50 ± 56)nV for the PTB comparison [1] and (72 ± 44)nV for the NPL comparison [4].

The BIPM Reference Groups of Standard Cells

The exploitation of an accurate Josephson-effect-based standard of emf is greatly aided by the availability of highly predictable and well-behaved standard cells. Such standard cells provide

1) references to maintain the emf standard between Josephson-effect runs ;

2) comparison cells for standard cell calibrations ; and

3) cross-checks on the performance of the Josephson-effect standard and cell calibration systems.

The apparatus and procedures described below fulfill these requirements with a precision of better than 10 nV.

The older of the two reference groups of cells, group V, consists of six saturated standard cells made at the PTB, Braunschweig. The standard cell enclosures that are at present commercially available are inadequate for our purposes. This is mainly because of temperature instability and pickup of alternating currents in the standard cell circuits. Alternating currents are known to change the emf's of standard cells [5, 6]. Our cells, which had already been stored at 20 °C for several years, were mounted in a prototype standard-cell air-bath enclosure in April, 1977. This enclosure has two temperature-regulated compartments, the outer one at 29 °C and the inner one at 30 °C. Thermistors are used as the temperature sensors in the two regulators and the temperature in the standard cell compartment is measured both with a thermistor and a platinum resistance thermometer. The former is measured with a built-in Wheatstone bridge for routine temperature measurements. The platinum thermometer, which is measured less frequently, provides a means of checking the thermistor. Two switches, one for polarity reversal and the other for cell selection, are mounted in the outer enclosure. Thermal shunts made of BeO, a good electrical insulator, are used to thermally anchor all wires traversing the walls of the enclosures. This helps to maintain a constant heat flow down the wires and, in the case of standard cell leads, to limit thermal emf's. Both the inner and outer temperature regulators employ dc bridge circuits and operational amplifiers. Usual precautions of careful grounding, twisted pairs of leads and the spatial separation of power leads from signal and standard cell leads were sufficient to eliminate any significant ac signals on the standard cell leads.

Figure 3 is a plot of the variations of the temperature of this enclosure about its reference temperature over the same 4-year period as in Figure 2. The temperature behavior may be roughly described as a linear increase of 1 mK/a upon which is superposed a sinusoidal annual variation of 1.3 mK amplitude. The measured short-term temperature drift rate has never exceeded 50 μK/d, except in July 1978 when a component failure occurred. We think that the linear variation is due to drift in components of the regulating circuit. For example, a drift rate in the relative value of the thermistor or the reference resistor in the regulating circuit of 5×10^{-5}/a would produce the observed linear temperature drift. We have found that the sinusoidal behavior is highly

correlated with the relative humidity in the building. Indeed, after correction for the linear drift, the difference between the enclosure temperature and its reference temperature is directly proportional to the relative humidity. The constant of proportionality is about 0.1 mK/degree of relative humidity.

Of the six cells in group V one is less "well-behaved" than the rest. Figure 4-A shows the behavior of the difference between cell V_4 and the mean of all six cells. This is one of the "good" cells. The initial, more rapid, drift rate over the first 350 days shown on the graph is typical of all six cells. The erratic behavior near day 1000 is due to the influence of other cells on the group mean. An example of this erratic behavior is given in Figure 5 which shows the behavior of cell V_6 with respect to the group mean. Spontaneous changes of 0.1 μV or more in the emf of V_6 were observed. The origin of these anomalies is not known. The absence of similar behavior in the cells that were compared with V_6 to obtain these data rules out the possibility that V_6 was charged or discharged during the measurements. In November 1980 cell V_2 was accidentally charged, resulting in a permanent increase of 60 nV in its emf. Because of the behavior of cell V_6 and of the discontinuity in the behavior of cell V_2, we usually treat the subgroup of four cells, group V_{1345}, as the reference group. Figure 4-B shows the behavior of cell V_4 with respect to the mean of subgroup V_{1345}. Comparison of Figures 4-B and 4-A clearly indicates the effect of the two deleted cells. Computer-drawn graphs of the type shown in Figures 4 and 5 are used for routine surveillance of our reference cells.

The second of our reference groups, group N, consists of five saturated cells also from the PTB, Braunschweig. These cells were placed in the enclosure after two separate groups of commercially made standard cells were tried and rejected. These commercial cells were subject either to discontinuous jumps in emf amounting to several tenths of microvolts or to high drift rates. The enclosure for group N is essentially a copy of that described by Cutkosky and Field [7], that is, it has inner and outer regulated enclosures and uses miniature platinum resistance thermometers for temperature control. The inner controller uses ac excitation, a transformer-ratio bridge and phase-sensitive detection. Some modifications of the Cutkosky design were made, the more important of which were the following : the use of a standard capsule-type platinum resistance thermometer for temperature monitoring ; the addition of a thermistor and its own external thermoregulated measuring bridge for routine temperature measurements ; and the addition of a thin-walled innermost cylinder containing the standard cells and intended to reduce convection currents and gradients.

The cells at present in the enclosure have been there since August 1980. They had previously been stored some six months at 30 °C, the nominal set point of group N, in an attempt to shorten the time apparently necessary for these cells to come to equilibrium after a large temperature change. Temperature measurements since October 1980 indicate a sinusoidal annual variation with an amplitude of 1.2 mK. Short-term temperature variations are less than 50 μK/d.

Standard Cell Comparison Systems

Two complete standard cell comparison systems are used in the BIPM emf-standard facility. A prototype system has been in operation since November 1976. It is located in the same screened room as the Josephson effect standard and it is used for the calibration of our reference cells. Based on experience with the prototype system, a second version was put into operation in October 1980 for routine standard cell comparisons. Most of the results given in this section, however, are those obtained with the prototype system. We shall come to results obtained with the second system later. Standard cell comparisons are made by measuring the difference in emf between two cells connected in series opposition. For our applications, the largest difference to be measured is just over 1.0 mV. This corresponds to the difference in emf between an unsaturated cell and a saturated cell at 30 °C. The difference is measured with a Julie Research Laboratories PVP 1001J, MOD 2 potentiometer with a VDR 307 J Kelvin-Varley divider. Because of a 12-step range on the most significant divider control, a full-scale reading of 1.2 mV is available on the lowest range of the potentiometer. The detector is a Guildline 9460 A photocell galvanometer-amplifier with a model 9461 B secondary galvanometer. The former was modified to increase the effective input impedance to about 15 kΩ at the usual gain levels.

The resolution obtained in the cell comparisons depends on a number of factors. For example, the ratio of rms thermal noise voltage to the square root of the detector bandwidth for a standard cell at 300 K having an internal resistance of 1 kΩ is about 4 nV/$\sqrt{\text{Hz}}$. Day-to-day variations in the level of mechanical vibrations in our laboratory lead to variations in resolution. In addition, some standard cells, particularly commercially made ones, have an intrinsic noise level above that attributable to the internal resistance. Under good observation conditions and with our best cells we can resolve a cell difference reading to within 3 nV. Indeed, the observed standard deviation of a single observation of the difference between a cell and its group mean is 3 nV for the comparisons of group V with group N. Under more usual conditions the resolution is about 5 nV. The acceptable upper limit is 10 nV.

Initial tests of the calibration of the potentiometer in the prototype system were made by direct comparison with a current-comparator-type potentiometer. The complete calibration of the potentiometer consists of calibrating the 120 kΩ Kelvin-Varley divider and the voltage ranges. A set of twelve 10 kΩ precision resistors was first calibrated by the substitution method using a current-comparator resistance bridge. These were then used to calibrate the divider by what is sometimes called the "k/n method". In this method n nominally equal fixed resistors are connected, say, in series and tapped at the k^{th} resistor, yielding a nominal ratio of k/n. The ratio correction is known from a previous calibration of the n resistors. The divider is connected in parallel with the n resistors. A detector connection from the divider output to the k^{th} resistor forms a bridge circuit. By varying k and n various ratios, hence divider settings, are obtained. The voltage ranges

were calibrated by measuring various fixed voltages on successive potentiometer scales, applying divider corrections. Divider linearity deviations are of the order of 3×10^{-6} of the full-scale setting at the settings of interest. This corresponds to an error of 3 nV for the comparison of saturated cells with unsaturated cells and less than 1 nV for the comparison of saturated cells at the same nominal temperature. The range uncertainties are about 4×10^{-6} of full scale on the scale of interest. This was obtained on the new instrument only after trimming of the range resistors.

Interconnections among the components of the standard cell comparison system are made with shielded and insulated cables consisting of a twisted pair of PTFE-insulated wires. The conductors are multistranded silver-plated copper. The shields are grounded and care is taken to avoid ground loops. Terminations are crimped copper lugs prepared from commercially available solder-plated lugs by removing the plating in hot hydrochloric acid. Thermal anchors are used in critical low-voltage circuits to minimize thermal emf's and their variations. Figure 6 shows a typical assembly. The crimp lugs are thermally anchored to, but electrically insulated from a thermal mass by a BeO washer. The assembly is held together with a Delrin*screw. A gold-plated copper nut maintains tight contact through the BeO with the thermal mass and acts as a locknut making it unnecessary to touch the BeO, a material which must be handled with caution. The materials used are not good absorbers of water and leakage resistances of 10^{12} Ω can be obtained with clean components.

The switches used in the standard cell comparator circuits are Stackpole series 100 rotary switches with coin silver contacts and terminals and Delrin and diallyl phthalate insulation. Leakage resistances of more than 5×10^{11} Ω are typical. Thermal emf's through the switches, including copper wire connections and low thermal emf solder joints, are generally below 3 nV.

Several sets of results allow us to evaluate the accuracy and precision of the prototype cell comparison system. Firstly we can examine the results of 25 Josephson-effect runs in which measurements were made on both saturated and unsaturated cells having a difference in emf, $E_u - E_s = \Delta E$, of about 1 mV. Here E_u is the emf of the unsaturated cell and E_s that of the saturated cell. This difference decreases at a rate of about 20 μV/a because of the drift of the unsaturated cells. Figure 7 is a histogram of the differences between ΔE(Josephson effect) deduced directly from Josephson-effect measurements and the value ΔE (Standard cell comparator) measured by the prototype cell comparator. The average difference is (6 ± 10)nV and the fact that it is nonzero is probably due to thermal emf's. Considering that the Josephson-effect measurement technique differs vastly from that of the cell comparator, we interpret these data as giving a good estimate of the accuracy of the cell comparator, albeit over a limited range and for large emf's. In this, as well as some of the following figures, the estimated mean, m, the estimated standard deviation, s_i, and the number of observations, n_o, are indicated. For purposes of comparison, corresponding normal distributions are also shown.

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* Delrin is the registered trade name of polyacetal.

Secondly, it should be borne in mind that each point in Figure 2 results from a set of Josephson-effect measurements on one cell and from 2 to 4 sets of cell comparisons made on the same day. Since the standard deviation of the mean for a Josephson run is 7.3 nV and is independent of cell comparisons and the average residual uncertainty from Figure 2 is 8.5 nV, the contribution to the dispersion in the data due to uncertainties in the cell comparisons is only about 4.4 nV.

One type of cell comparison design, or measuring sequence, that we often use in the comparisons of our reference groups involves the pairwise comparison of each cell of one group with two cells of the second group in such a way that each cell of each group participates in two comparisons. We call the algebraic sum with alternating signs of the measured differences the residual of the comparison. In the absence of error its value is zero. The residuals provide an estimate of the repeatability of the cell measurements.

Figures 8, 9 and 10 show histograms of the residuals obtained in the comparisons of three different cell groups with reference group V at various times. Figure 8 gives the residuals of 180 comparisons of a group of unsaturated cells from January 1978 to June 1980. Figure 9 gives the residuals of 46 comparisons with a new group, group T, of five unsaturated cells in a new enclosure having a built-in polarity switch. These results cover the period from July 1980 to June 1981. Figure 10 represents the present situation at BIPM. Shown here are the residuals for all of the 31 comparisons in 1981 between group N of five high-quality saturated cells and group V.

New Standard Cell Comparison Facility

The new standard cell comparison facility was designed and constructed using the results of several years of experience with the prototype system. It replaced the old standard cell comparison system described by Romanowski [8]. The new facility is located in the laboratory that contains the screened room for the Josephson effect and the chamber for the standard cell oil bath, the three systems being separated by only a few metres.

To minimize the effects of the rather high level of electromagnetic interference in this location, a screened room was built to house the new facility. The room is 3.4 m long, 2 m wide and 2 m high. The walls, ceiling and floor are made of 2 m × 1 m sheets of 1-mm-thick galvanized steel. Joints between sheets overlap by 20 to 30 cm and are reinforced by steel bands. Sections were joined in place with rivets. The door is made of two galvanized steel sheets welded to a tubular steel frame. The door is sealed by phosphor bronze leaf springs of the type used on commercial screened rooms. The seal is made against a copper jamb. Mains power is fed into the room through a low-pass filter. The mains power line itself is connected to an emergency generator which provides power in case of an electrical failure. To minimize induced ac at power

frequency in sensitive standard cell circuits, the power lines inside the room are placed in steel tubes which are positioned away from sensitive components. Two shelves are provided to take the standard cell enclosures. These are fixed to wooden beams solidly embedded into the outside walls and covered with galvanized steel. The screened room is located on a balcony of the laboratory and its floor is not stable enough to take the photocell galvanometer-amplifiers. A galvanometer mount was fixed to a solid building wall in the following manner. First a polycarbonate plate was cemented into the stone wall. Six large threaded holes in the 25-mm-thick plate take the short bolts which fix the mount to the plate. The bolts are short enough to prevent an appreciable antenna effect. Next the galvanized steel sheet, part of the screened room wall, was put into place. Over this was placed a sheet of lead foil 3 mm thick in an attempt to level out the effect of irregularities in the wall and to lessen the transmission of vibrations. This mount has proven to be effective in decoupling the floor vibrations from the photocell galvanometer-amplifier.

Several feed-throughs into the screened room are required for standard cell and thermometer leads. These are made of hollowed-out connector shells soldered to the screened room walls and closed, when not in use, by fine-threaded caps. Feed-throughs are also provided for two shafts extending down to the oil bath below the screened room. These shafts turn the rotary polarity and selector switches for oil-bath cells which are compared with the cells of group N in the screened room. When measurements on oil-bath cells are not being made, removable sections of the shafts are withdrawn and the feed-throughs are capped.

In the screened room, long runs of signal-carrying cables between cell enclosures and the cell comparator are lead through aluminum conduits to shield them from drafts. Particular care was taken in the layout of ground leads. Any grounded component is connected to ground by one path only, avoiding loops. A separate ground scheme, in which the ground lead for every component is connected in a parallel fashion to a single point on the ground bus, is used for the sensitive parts of the measuring system, such as the potentiometer, the galvanometer-amplifier, the secondary galvanometer and cable shields. A second separate ground line, also providing parallel ground leads to individual components, is reserved for the chassis grounds of commercial standard cell enclosures. This was found to be important because of significant ac in the chassis leads to ground. Finally a third ground is provided for the mains plugs. The three ground leads merge at one point only, where they are connected to a single ground line.

The standard cell comparison equipment used in the new installation is of the same type as the prototype system described above. At the present time, the reference cells are those of group N, also described above.

Measurement Procedures

The first piece of information needed for the calibration of standard cells is the value of the working reference group in terms of the Josephson emf standard. Josephson-effect measurements are made in

groups of two or three runs over a period of about two weeks. This schedule was adopted to make the most economic use of liquid helium. The normal time between successive groups of measurements is about ten weeks. One Josephson-effect run occupies the working day of one person.

Before and after Josephson-effect runs, standard cell intercomparisons are made between group V, which contains the cell measured by the Josephson effect, and a second group of reference cells. This comparison is made with the prototype cell comparator. Presently the second reference group is group N, consisting of five saturated cells.

A typical standard cell comparison design is given in Table II for the case of a 5-cell group, N, versus a 6-cell group, V. In this case cell N_2 is compared twice in order to maintain a partial symmetry. In the comparison of any pair of cells we always make measurements in both polarities, that is, with the cells connected together once by their positive poles and once by their negative poles. The potentiometer current is appropriately reversed. We assume that a thermal emf e_i is associated with each pair of cells. Measurements are made in the order in which they appear in the table.

Temperature corrections to the cells' emf's are based on measurements of the thermistors in the enclosures. The platinum resistance thermometers are normally used only as cross-checks. Care is taken in the design of the thermistor bridges to assure that the measuring current in the thermistor has adequate long-term stability or is reproducible by adjustment. This is important since the thermistor measurements are done at a single current value (approximately 20 μA) and self-heating effects due to measuring current changes could be significant. In the case of unsaturated cells, no temperature corrections are applied. In early Josephson-effect work at BIPM unsaturated cells were sometimes used because of their low temperature coefficient of about - 1 μV/K. They were used in a commercial cell enclosure the temperature stability of which was, for our purposes, insufficient for use with saturated cells. In 1980 a new group of commercially-made unsaturated cells was put into a prototype enclosure for experimental purposes. This is group T.

From these data we calculate the difference between each cell and its group mean and the difference between group means, corrected for temperature. Since the value of one cell is known in terms of the Josephson standard, i.e., in $V_{76\text{-BI}}$, the values of the other cells can be calculated in terms of $V_{76\text{-BI}}$.

In the intervals between Josephson-effect measurements, the unit of emf is maintained by reference subgroup V_{1345} the behavior of which is shown in Figure 2. Weekly comparisons between group V and group N, the "working reference group", are usually done with the prototype comparison system but, for purposes of verification, they are occasionally done with the new system.

Routine Calibration of Standard Cells

So far we have described the realization of the BIPM standard of emf and the calibration of our reference standard cells. The results we obtain on our reference cells may be considered as those obtained under optimum conditions inasmuch as they are obtained on high-quality cells in very well-regulated enclosures and the cells are not transported. In contrast, the cells sent to BIPM for calibration or as part of an international comparison are most often cells of commercial quality in commercially-made enclosures or in no enclosure at all and have, of course, been transported. In the following sections we describe the material and procedures used in these calibrations and some of the results obtained.

Transportable Standard Cell Enclosures

There is no doubt that the most accurate way of comparing emf standards between laboratories is by means of direct comparisons of the Josephson effect. However this is a time-consuming and costly process which until now has only been done experimentally. The next-best alternative is to transport standard cells in their own enclosures.

Cells in a thermoregulated enclosure form a combined emf-temperature transfer standard. Since the enclosure has its own built-in thermometer one does not need to know the temperature of the cells on IPTS-68 (t_{68}) but only the difference between the enclosure's present temperature and its reference temperature. The built-in thermometer is usually a thermistor. By contrast, the calibration of standard cells in an oil bath requires an accurate measurement of t_{68}. For example, for cells at 20 °C where the temperature coefficient of a standard cell is about - 40 μV/K a difference of 1 mK in temperature scale between two laboratories leads to a 40 nV difference in emf assigned to a cell.

The second advantage of the use of thermoregulated enclosures for the transfer or comparison of emf standards is the avoidance of a long lag time in the reestablishment of equilibrium of the emf of a saturated cell. A long lag time resulting from an abrupt 10 K increase of cell temperature may be seen in the data of Figure 2.

Inadequacies of Commercial Standard Cells and Enclosures

Although they can in principle lead to better comparisons of emf standards, standard cells and enclosures, especially of the commercial type, unfortunately have a number of defects.

1) The first defect is that the cells provided with commercial enclosures are sometimes of mediocre quality either because of large drift rates of emf or because of spontaneous unpredictable discontinuities in the emf. In 1979 we tested 24 commercially made standard cells of recent construction in a prototype enclosure with the hope of selecting good-quality cells from the group. Of the 24 cells only six had a long-term drift rate with an absolute value of less than 1 μV/a. Ten cells had deviations of over 0.1 μV from a linear fit to the short-term behavior of the emf as a function of time. This is in great contrast with the cell behavior shown in Figure 4-B.

2) A common defect in commercial standard cell enclosures is the presence of spurious alternating voltages on the standard cell leads. These voltages are usually at twice the mains frequency. Low frequency alternating currents are known to change cell emf's [5, 6]. The magnitude of the change decreases with frequency and increases with cell age and internal resistance. Jennings and O'Connor [5] report that, in a typical case, a 9-year-old cell of 1380 Ω internal resistance changed emf by 0.73 μV when 1 μA rms of alternating current at 50 Hz passed through it. We have often observed alternating voltages between cell terminals and ground. In one severe case we observed 30 V rms on the terminals of cells in a commercial enclosure.

3) A less serious but nevertheless annoying problem is the inductive coupling between the heater circuit and the standard cell leads which causes spurious deflections of the null detector as the heater cycles.

4) For the highest precisions the commonly-used commercial enclosures are not stable enough in temperature. These enclosures have short-term stabilities of the order of 1 mK for, say, several days, which corresponds to a stability of about 55 nV in the emf if no significant gradients are present. It is not uncommon to observe abrupt temperature changes between successive comparisons. In the absence of continual temperature monitoring the time of the change is unknown, making it difficult to know if the cells are in thermal equilibrium.

5) Occasionally there are errors in the calibration of the thermistor bridges in commercial enclosures. We have deduced apparent errors of some tens of percents in the calibration of the temperature indication dial of some enclosures. In one extreme case we found that the dial indicated temperature changes just opposite to the real ones and that the potentiometer control was wired in backwards. It is worth recalling that linear secular variations of the measuring thermistor or its associated reference resistor would not necessarily result in a loss in attainable accuracy. It would be indistinguishable from a linear drift in the cells.

In view of the problems encountered with commercial cells and enclosures, we use non-commercial standard cells and in our own carefully-made enclosures. This solution is not easily adopted by some laboratories and so we offer the following comments and suggestions to those who have no alternative to using commercial cell enclosures :

1) The results we obtain with non-commercial standard cells from the PTB, Braunschweig demonstrate that it is possible to make high-quality standard cells. Such cells may be expensive and difficult to obtain commercially. An alternative is to select the best cells from a large group of ordinary commercial cells. From our experience with the group of 24 cells mentioned above, this solution is time-consuming and the results are mediocre but are an improvement over unselected cells.

2) To avoid problems associated with alternating currents on standard cell leads we have adopted the procedure, when possible, of operating commercial enclosures from 12.5 to 13 V power supplies in

parallel with a battery connected to the "12 V standby" terminals often provided on the enclosures. The power supplies must be carefully grounded and tested for residual ac. In general, we have observed changes of a few mK between the temperature maintained under dc operation and that obtained under mains operation. Thus one should not operate an enclosure from the mains and, for the time required for a cell comparison, operate it from a dc supply without previous verification that this operation does not change the temperature.

3) If the requirements are such that short-term temperature stability of better than 1 mK is necessary the user must build his own standard cell enclosure. A detailed report on the construction and performance of particularly simple enclosures of sufficient stability is in preparation [9].

4) Verification of the accuracy of temperature corrections applied to commercial enclosures can be made by a procedure described below. This is based on the usual spontaneous variations of the enclosure temperature. A more precise solution is to modify the temperature controller bridge by connecting shunt resistors into the circuit. By inducing changes of up to about 10 mK in the operating temperature, one could measure the temperature coefficient of the cells directly as a function of the position of the thermistor bridge dial.

5) For some purposes the scale of the temperature indicator dial of commercial enclosures is too coarse. A simple method of obtaining greater resolution in temperature is to read the thermistor bridge output voltage at the engraved divisions to each side of the null position. A linear interpolation can then be made to obtain the temperature corresponding to the voltage read at zero bridge current.

Cell Comparison and Data Reduction Procedures

The comparison of cells in the oil bath is done in much the same way as that for cells in enclosures so here we limit the explanation to the latter. The measurement design we have chosen to use is very simple. One cell of group N, the emf of which is known in terms of the Josephson standard, is compared with each cell of the group to be calibrated. Each cell is compared to the standard in both polarities. Temperature measurements are made before and after the emf measurements. Typically, calibrations are made once a week for about 12 weeks. The reference cell information is obtained from comparisons of the type indicated in Table II which are done weekly. These are occasionally done with the routine comparison system as a check on instrumentation errors.

Measurement results are entered immediately into a Hewlett-Packard 85 desk computer which calculates cell values based on provisional estimates of the reference cell. More importantly, the data are stored in a magnetic tape file which will eventually be used in the definitive calibration results. Final calibration results are calculated only after a final Josephson-effect measurement is done so that the emf's of the reference groups are deduced by interpolation of the data and never by extrapolation of the cell behavior. The program for the analysis of the calibration data calculates various statistical information including a linear least-squares fit to the emf's as a function of time. In particular, the uncertainty of the drift rate allows us to evaluate its

significance. The previously mentioned check on the calibration of the temperature indication dial of the cell enclosure is included in the analysis. This check consists of making a linear least-squares fit to the group mean emf, already corrected for temperature using a standard formula [10], as a function of the indicated temperature. In addition, the data are plotted. A word of caution should be added on this technique. Monotonic temperature changes with time could lead to erroneous conclusions about the temperature bridge calibration if, during the same time period, the cells drift monotonically. In practice, the observed temperature usually changes in both directions even for periods as short as several months.

Uncertainty Due to the Transfer of Standard Cells

In the comparison of emf standards between two laboratories the largest contribution to the uncertainty is due to the instability of the transfer standard cells. It is very difficult to estimate this uncertainty for the general case because it strongly depends upon the properties of individual cells as well as upon their handling.

In an attempt to evaluate the stability of standard cells as transfer standards we have surveyed data from four international comparisons of emf standards involving 37 groups of cells from 1970 to 1979 [11]. In these comparisons each laboratory reported values of its cells, typically four to six in number, in terms of the laboratory's national standard, before and after the cells were sent to BIPM for comparison. For each cell the difference between the emf measured in the other laboratory after the comparison at BIPM and the emf determined by the laboratory before the comparison is given in the histograms of Figures 11 to 14. We distinguish between cells measured in an oil bath at 20 °C, which in general were not shipped under thermoregulation, and cells in air-bath enclosures at 30 °C which were. Individual cells sometimes change by unusually large amounts, for example several microvolts. These cells are usually eliminated from the comparison data at the request of individual laboratories. We have excluded such "outliers" in these figures. This practice makes statistical interpretation of the data, which are usually scanty, even more difficult.

Figures 11 and 12 summarize the observed changes in cells, measured at 20 °C, participating in the 1970 and 1973 intercomparisons. One outlier was eliminated from Figure 11 and two from Figure 12. The large dispersions of the results reflect the difficulty of assigning an uncertainty due to the transportation of cells. Note that in these figures the abscissas are in units of μV.

Figure 13 shows the behavior of 71 cells in air-bath enclosures at 30 °C which participated in the four comparisons. Figure 14 gives the behavior of 35 cells in air enclosures sent by European national standards laboratories.

These figures confirm two widely held views, namely 1) that cells in transportable thermoregulated enclosures are more stable transfer standards than cells transported under no thermal regulation ; and 2) cells which travel farther are likely to undergo larger emf changes.

The data in these figures do not take into account secular drifts in the cells for the time between measurements. However the mean changes do not differ greatly from zero, especially when compared with the corresponding standard deviations. This implies that it is difficult to justify interpolation of cell values, in terms of the individual laboratory standards, to the mean date of the BIPM comparison. Indeed, Figure 15 illustrates the case of a standard cell comparison between BIPM and a national laboratory in which a linear interpolation was clearly not justified. The solid line represents the linear least-squares fit made to the mean of the group as a function of time based on the measurements at BIPM. The dashed line is drawn through the two points supplied by the participating laboratory. The difference in the slopes is clear. For lack of other information, a simple mean of the two laboratory calibrations may be more desirable for calculation of the cell values in terms of the laboratory standards. Of course, proper timing of the measurements makes the results of the two methods, linear interpolation and mean, identical.

We assume that the dispersion in the observed changes in the cells is an estimate of the uncertainty attributable to their stability as transfer standards. The data in Figures 11 through 14 then lead us to conclude that in the most favorable case, namely, when enclosures are used, the expected uncertainty in international comparisons of standards of emf is 0.5 μV.

A somewhat different transfer technique using standard cells has been developed by the National Bureau of Standards (NBS) and is known as the Volt Transfer Program [12]. In this technique, a commercial-type group of cells and enclosure belonging to NBS is sent to the outside laboratory. The cells are measured in terms of the laboratory's emf standard and returned to NBS for re-calibration in terms of the NBS unit of emf. The uncertainty of such a comparison is estimated by NBS to be about 0.14 μV.

These techniques were used in international comparisons in 1971 and 1972 by the NBS to compare values of 2e/h obtained in national standards laboratories [12]. Notwithstanding the increased distances involved, the assumed uncertainty for a single transfer was 0.14 μV. In all, the comparisons involved 36 cell measurements. Of the 36 cells 5 were rejected. In addition, for one group of 4 cells the emf variations before and after the transfer were not reported because of nonlinear drift. For the remaining 27 cells we calculate an average change in emf of a group before and after shipment from NBS of - 0.02 μV and a standard deviation of 0.18 μV. This is not inconsistant with our estimated uncertainty of 0.5 μV because we treat the cells individually, not as groups, and our criteria for eliminating outliers are not the same.

In one other reported intercomparison [13], five regional standards laboratories in a relatively small geographic area intercompared units of emf using the NBS techniques. Redundant measurements allowed a calculation of the standard deviation for a single comparison. This was

0.35 μV, in good agreement with our estimates for the case of reduced travel distances.

We would admit that in isolated cases and with special equipment it may be possible to obtain uncertainties of less than 0.1 μV in the international comparison of emf standards by transferring standard cells. However the published data cited here indicate that such accuracy cannot generally be realized.

Uncertainties in BIPM Measurements and in International Comparisons

In this section we summarize all of the contributing uncertainties in the international comparison of units of emf by the transport of commercial-quality cells and enclosures to the BIPM. Figure 16 indicates the principal steps in a comparison. From Table I, the uncertainty in the reference cells of the BIPM with respect to the Josephson-effect standard is 24 nV. The additional uncertainties are indicated in Table III. The following comments are relevant.

The uncertainty of the measurements done at the BIPM are based on observations of better-quality commercial cells in air enclosures. About 50 % of the enclosures and cells would exhibit worse behavior.

The uncertainty due to the standard cell comparator system is proportional to the difference in emf between our reference cells and the cells to be compared. In the case considered here, this difference is of the order of tens of microvolts or less. In the range of differences from 100 to 1 100 μV, this uncertainty could be as much as 10 nV.

If there is reason to believe that the rates of change of emf observed at the BIPM are part of the permanent behavior of the cells and not just temporary results of transport, then the uncertainty in the measured drift rate used in the extrapolation should be taken into account. The uncertainty is a hyperbolic function [14] of the elapsed time between the central date of the BIPM measurements and those of the other laboratory. The estimate in Table III assumes elapsed times of 10 weeks before and after the BIPM central date. In this estimate we assume average drift rates and uncertainties based on our observations on "better" cells.

The uncertainty due to the stability of standard cells is seen to be an order of magnitude greater than the next-largest single uncertainty. This transfer uncertainty is two orders of magnitude greater than the reproducibility obtainable by careful measurements of high-quality stationary standard cells.

Conclusions

The results presented here show that our cells, enclosures and measurement techniques well-fulfill the three requirements enumerated above, namely, the maintenance of the emf standard between Josephson-effect runs, the provision of comparison cells and, by virtue of their excellent reproducibility, the existance of a cross-check on the Josephson-effect and cell-comparison systems.

Results of comparisons by national standards laboratories show that the performance of standard cells as transfer standards is about one hundred times worse than their performance as stationary laboratory standards. New transfer standards which are more stable than presently used standard cells, but cheaper and more convenient than the Josephson effect, are needed. The high levels of precision in emf measurements, demonstrated by our results, together with the temporal stability of the Josephson effect allow one to relax to some extent the qualities required of an improved transfer standard of emf. Predictable relative changes of several parts in 10^7 per day could be tolerated.

Acknowledgment

The authors wish to thank the PTB, Braunschweig for making their standard cells available to the BIPM.

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FIGURE CAPTIONS

Figure 1. Histogram of the measured values of the standard deviation of the mean of 68 Josephson-effect runs for 1978 through 1981. The mean value is 7.3 nV.

Figure 2. Behavior of the mean of four saturated cells of group V_{1345} as determined by Josephson-effect measurements and standard cell comparisons. Each datum represents a Josephson-effect run using either a saturated cell or an unsaturated cell. The straight line results from a linear, unweighted, least-squares fit to the data after June 1979. The slope of the line is 0.20 nV/d. The average residual uncertainty of an observation with respect to the line is 8.5 nV.

Figure 3. Variations of the temperature T of group V with respect to the reference temperature from 1978 through 1981.

Figure 4-A. Behavior of cell V_4 with respect to the mean of all six cells. This is considered to be a "good" cell.

Figure 4-B. Behavior of cell V_4 with respect to the mean of the subgroup V_{1345} of four "good" cells.

Figure 5. Behavior of cell V_6 with respect to the mean of all six cells. This cell exhibited anomalous spontaneous changes in emf in December 1978 and June 1980.

Figure 6. Detail of a thermal anchor using a BeO washer.

Figure 7. Histogram of the differences between ΔE(Josephson effect), the difference between the emf of an unsaturated cell and a saturated cell, both measured by the Josephson-effect apparatus, and ΔE(Standard cell comparator), the same difference measured using the standard cell comparator, for the 19 measurements of this type in Figure 2 and six measurements made in 1977. The average difference ΔE(Josephson effect) - ΔE(Standard cell comparator) is (6 ± 10)nV. The curve is a fitted normal distribution (see text).

Figure 8. Histogram of residuals for 180 comparisons between group BINS of unsaturated cells and group V from January 1978 to June 1980.

Figure 9. Histogram of residuals for 46 comparisons between group T of unsaturated cells and group V from July 1980 to June 1981.

Figure 10. Histogram of residuals for all 31 comparisons between group N of saturated cells and group V from April to December 1981.

Figure 11. Changes in the emf's of transfer cells determined by the national standards laboratories, in terms of their own emf standards, after round-trip shipment to BIPM for the 1970 comparison. The cells were measured in oil baths at 20 °C. One "outlier" was eliminated from the data.

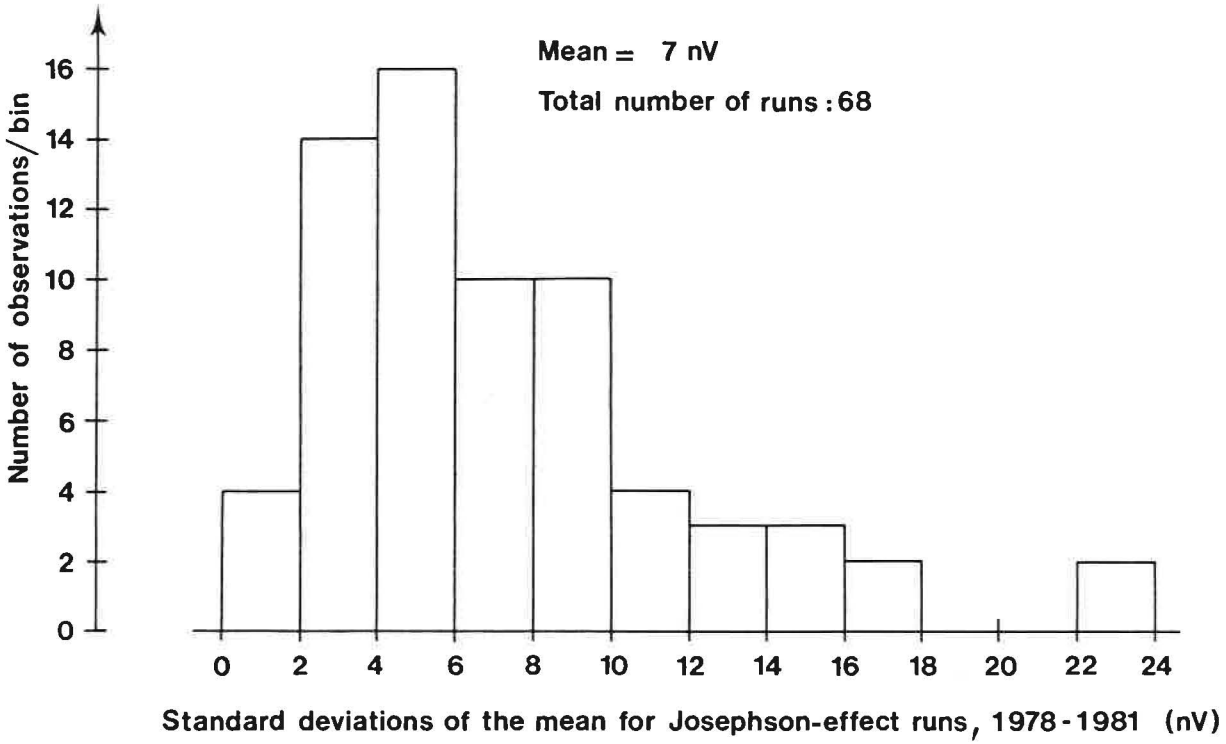
Figure 12. Changes in oil-bath cells at 20 °C for cells participating in the 1973 comparison. Two "outliers" were eliminated from the data.

Figure 13. Changes in air-enclosure cells participating in the 1970, 1973, 1976 and 1979 comparisons. One "outlier" was eliminated.

Figure 14. Changes in air-enclosure cells from European laboratories participating in the 1973, 1976 and 1979 comparisons.

Figure 15. Values of the mean emf of a group of cells measured by the BIPM and by the laboratory originating the comparison. This illustrates the difficulties of extrapolating cell emf's after transporting the cells.

Figure 16. Schematic diagram of the principal steps and sources of uncertainty in the comparison of units of emf with an external laboratory by transport of standard cells.



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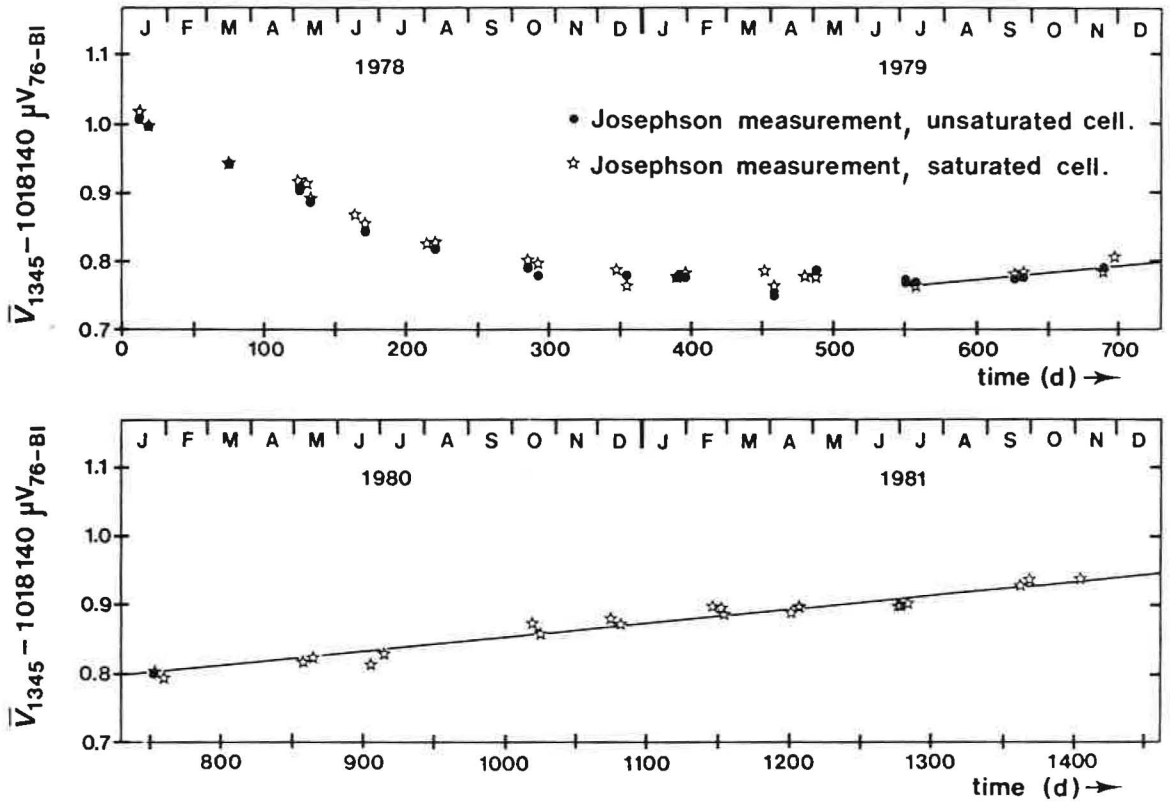
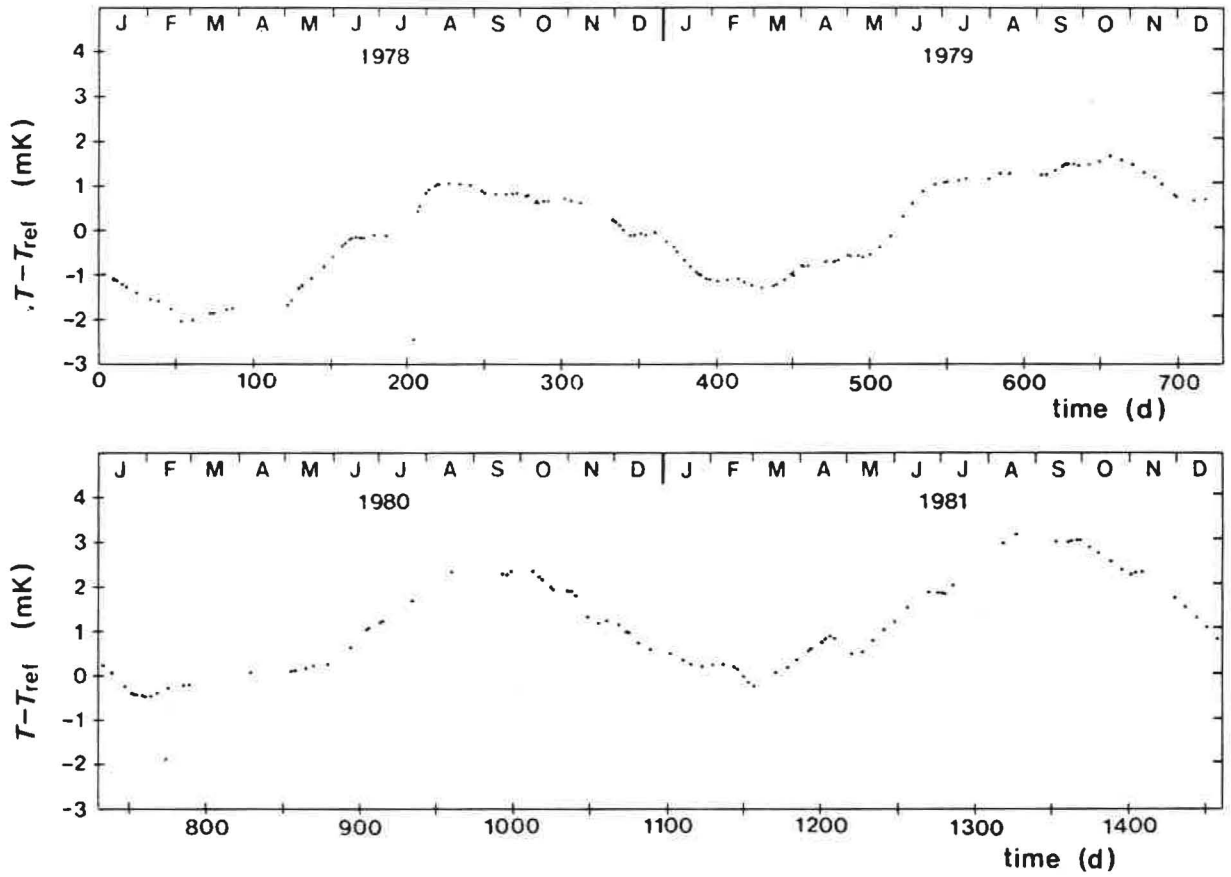


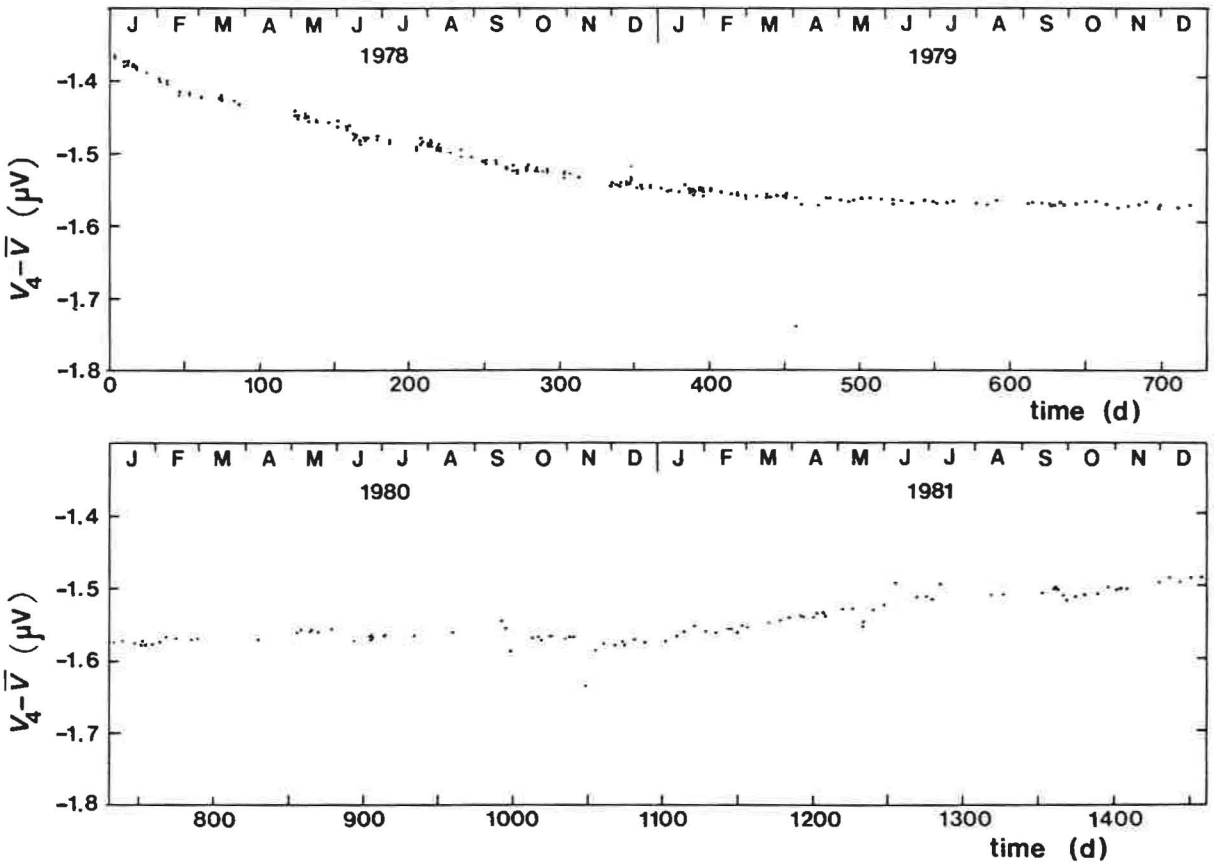
Josephson-effect determination of the mean emf of a subgroup of 4 cells of group V, 1978-1981

Fig. 2.- Behavior of the mean of four saturated cells of group V_{1345} as determined by Josephson-effect measurements and standard cell comparisons. Each datum represents a Josephson-effect run using either a saturated cell or an unsaturated cell. The straight line results from a linear, unweighted, least-squares fit to the data after June 1979. The slope of the line is 0.20 nV/d. The average residual uncertainty of an observation with respect to the line is 8.5 nV.



Temperature of group V, with respect to its reference temperature, over 4 years time

Fig. 3.- Variations of the temperature T of group V with respect to the reference temperature from 1978 through 1981.



emf of cell 4 , a "good" cell , with respect to the mean of the 6 cells of group V, 1978-1981

Fig. 4-A.- Behavior of cell V_4 with respect to the mean of all six cells. This is considered to be a "good" cell.

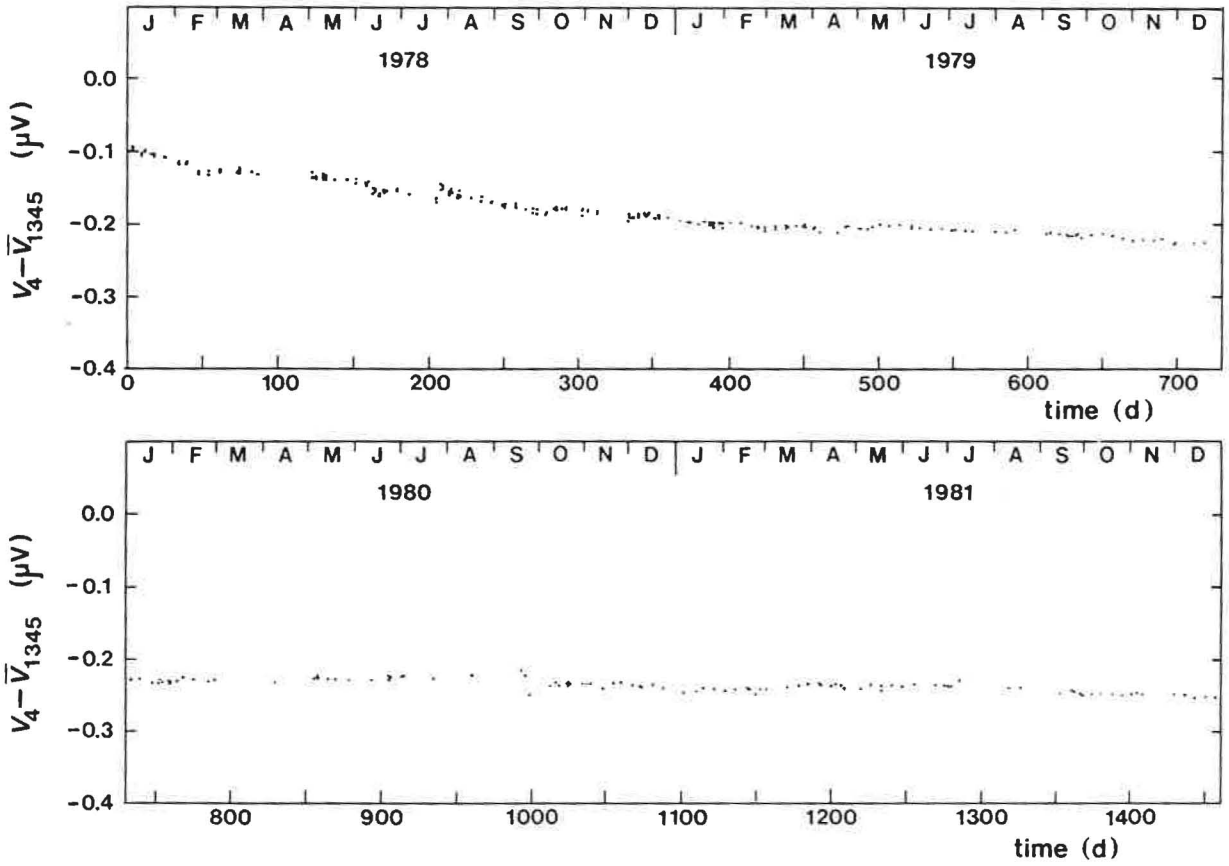
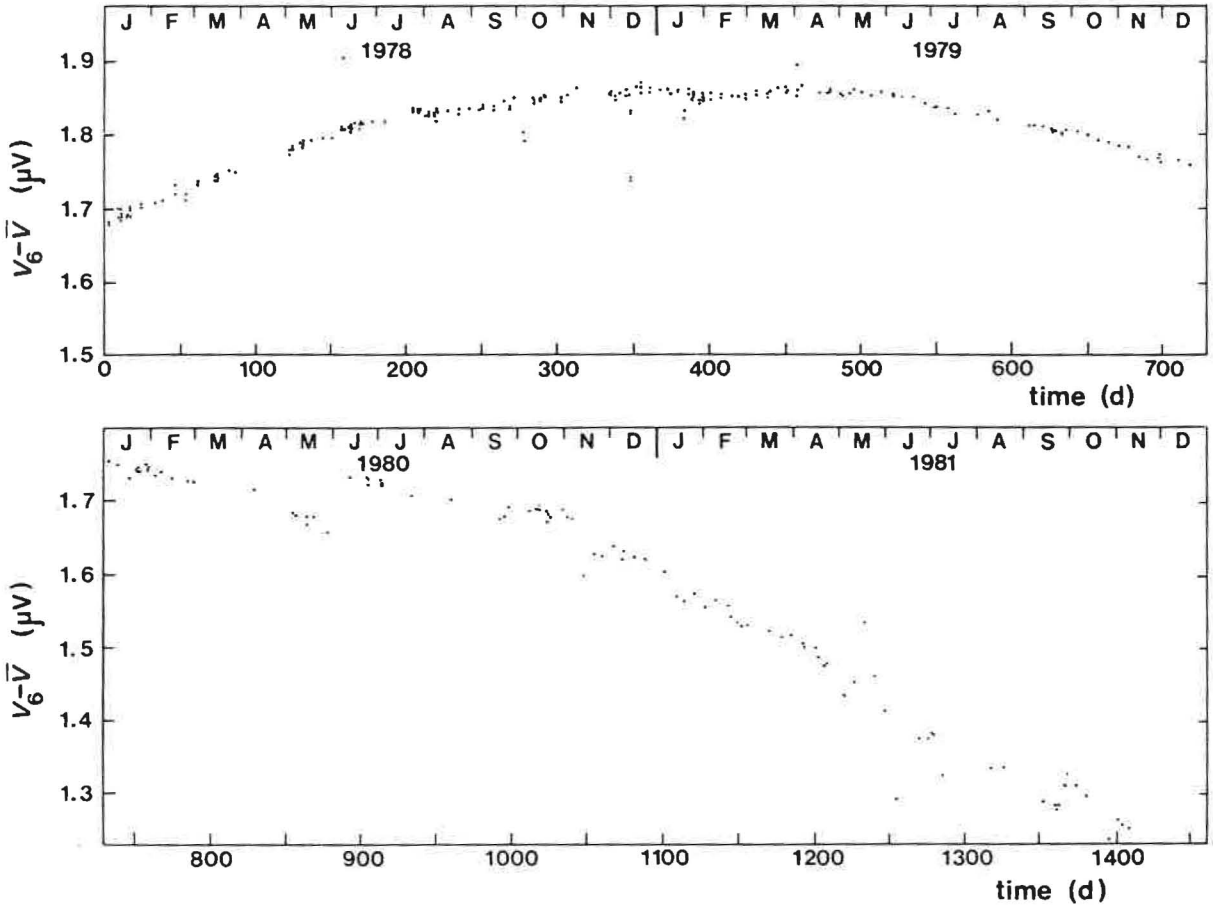


Fig. 4-B.- Behavior of cell V_4 with respect to the mean of the subgroup V_{1345} of four "good" cells.



emf of cell 6, a "bad" cell, with respect to the mean of the 6 cells of group V, 1978-1981

Fig. 5.- Behavior of cell V_6 with respect to the mean of all six cells. This cell exhibited anomalous spontaneous changes in emf in December 1978 and June 1980.

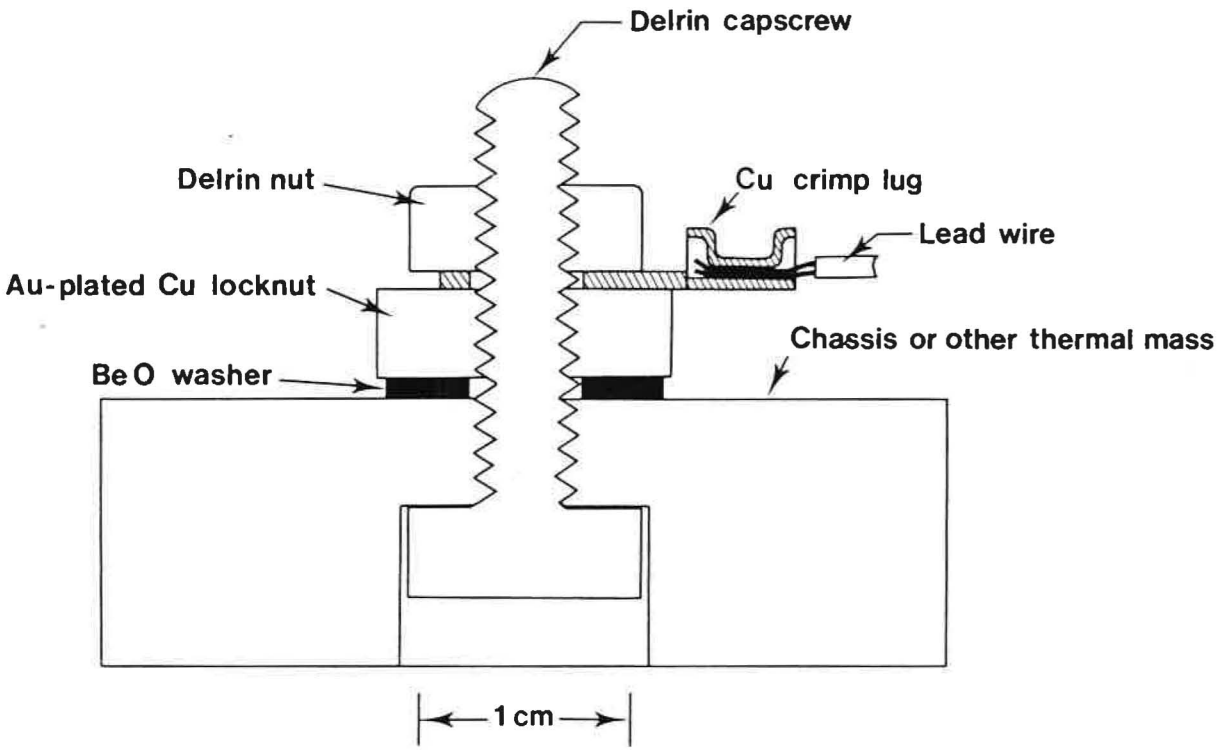
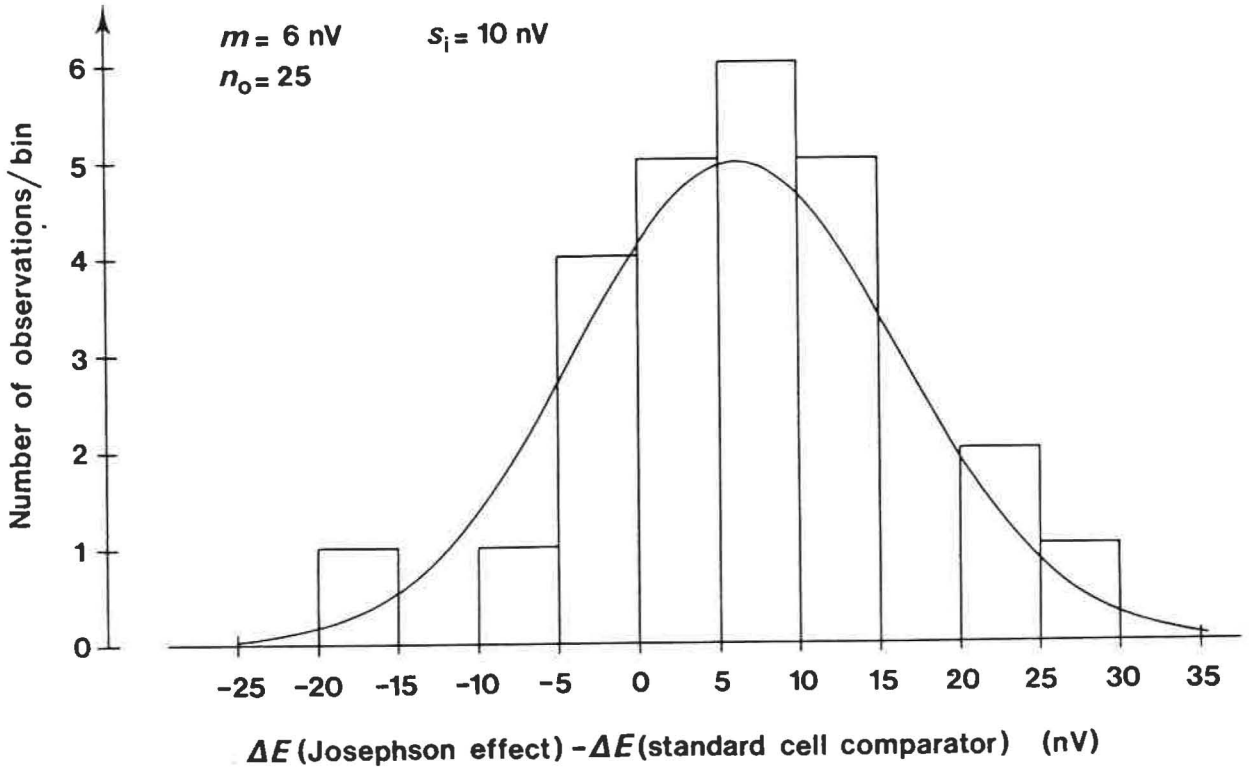


Fig. 6.- Detail of a thermal anchor using a BeO washer.



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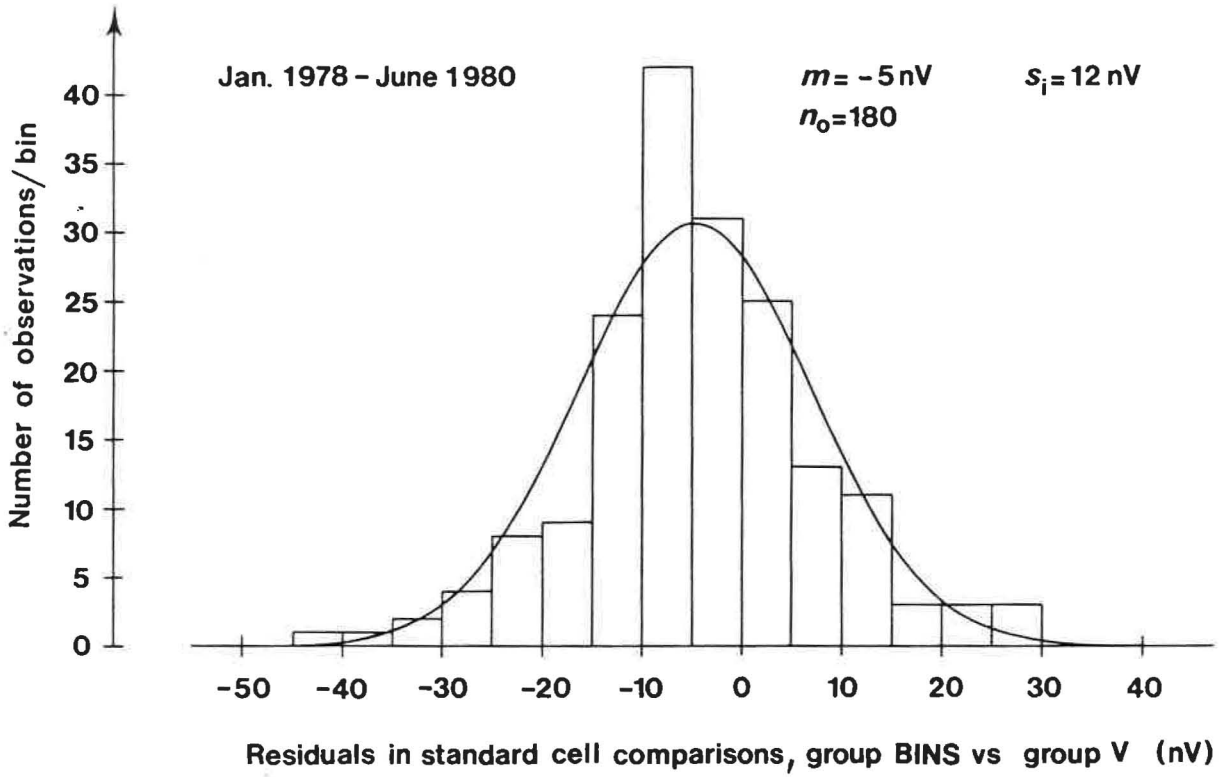

Fig. 8.- Histogram of residuals for 180 comparisons between group BINS of unsaturated cells and group V from January 1978 to June 1980.

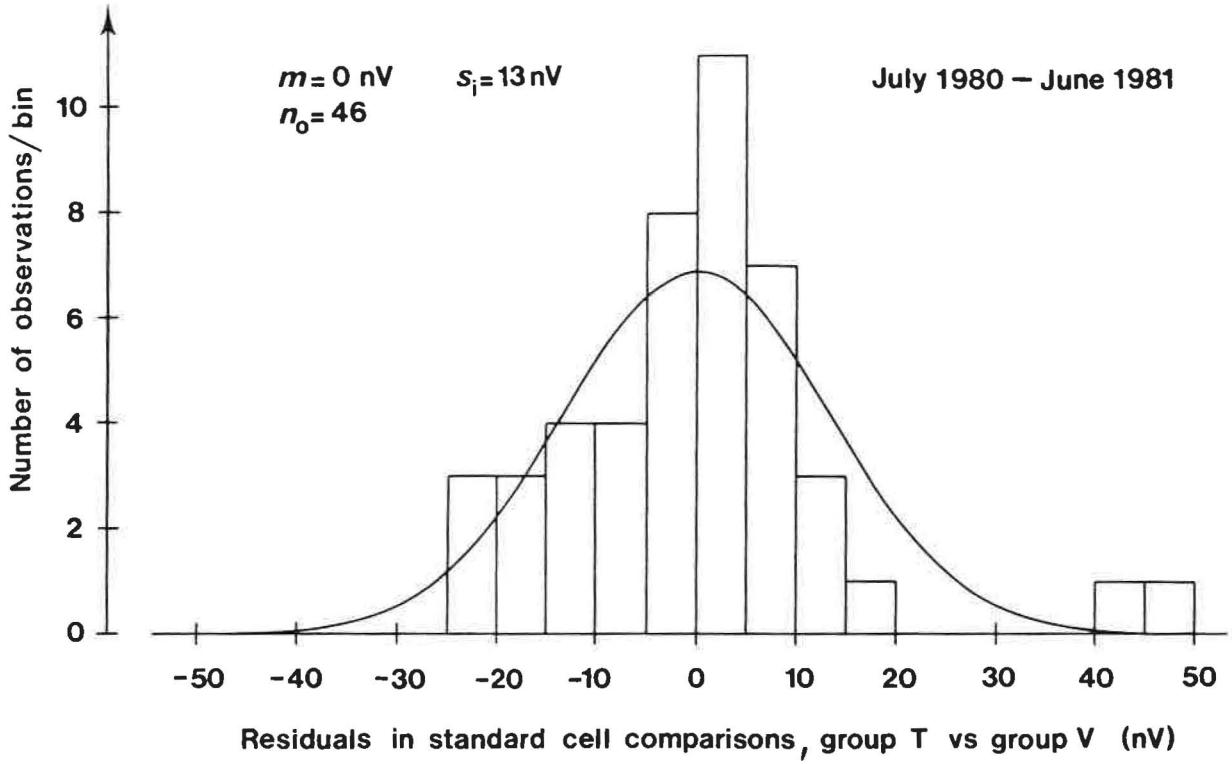

Fig. 9.- Histogram of residuals for 46 comparisons between group T of unsaturated cells and group V from July 1980 to June 1981.

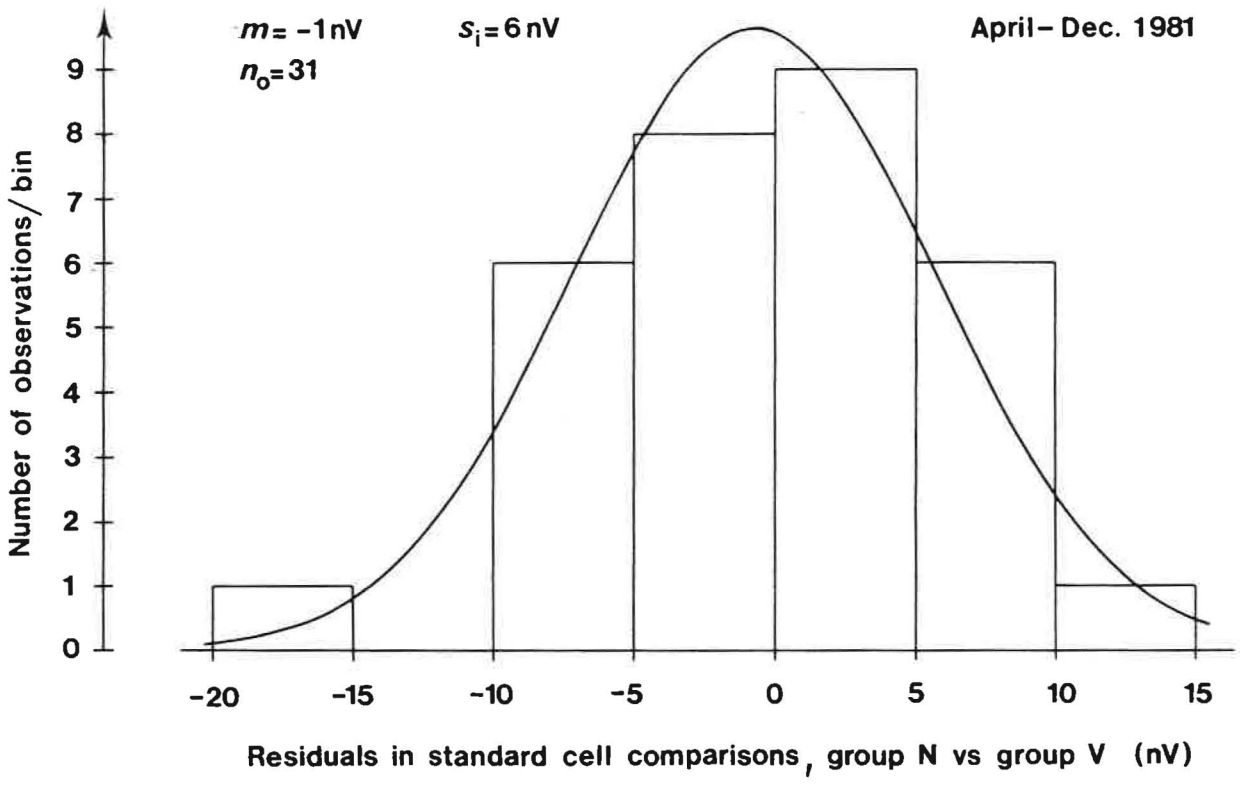

Fig. 10.- Histogram of residuals for all 31 comparisons between group N of saturated cells and group V from April to December 1981.

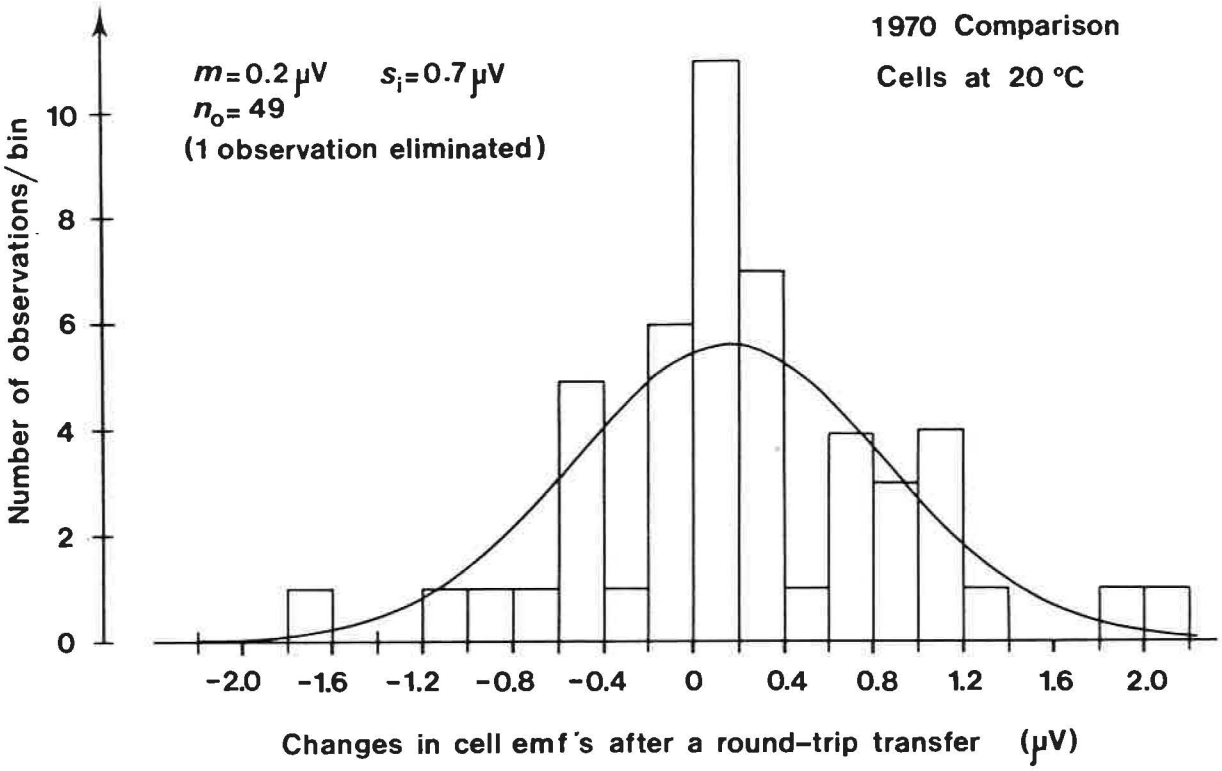

Fig. 11.- Changes in the emf's of transfer cells determined by the national standards laboratories, in terms of their own emf standards, after round-trip shipment to BIPM for the 1970 comparison. The cells were measured in oil baths at 20 °C. One "outlier" was eliminated from the data.

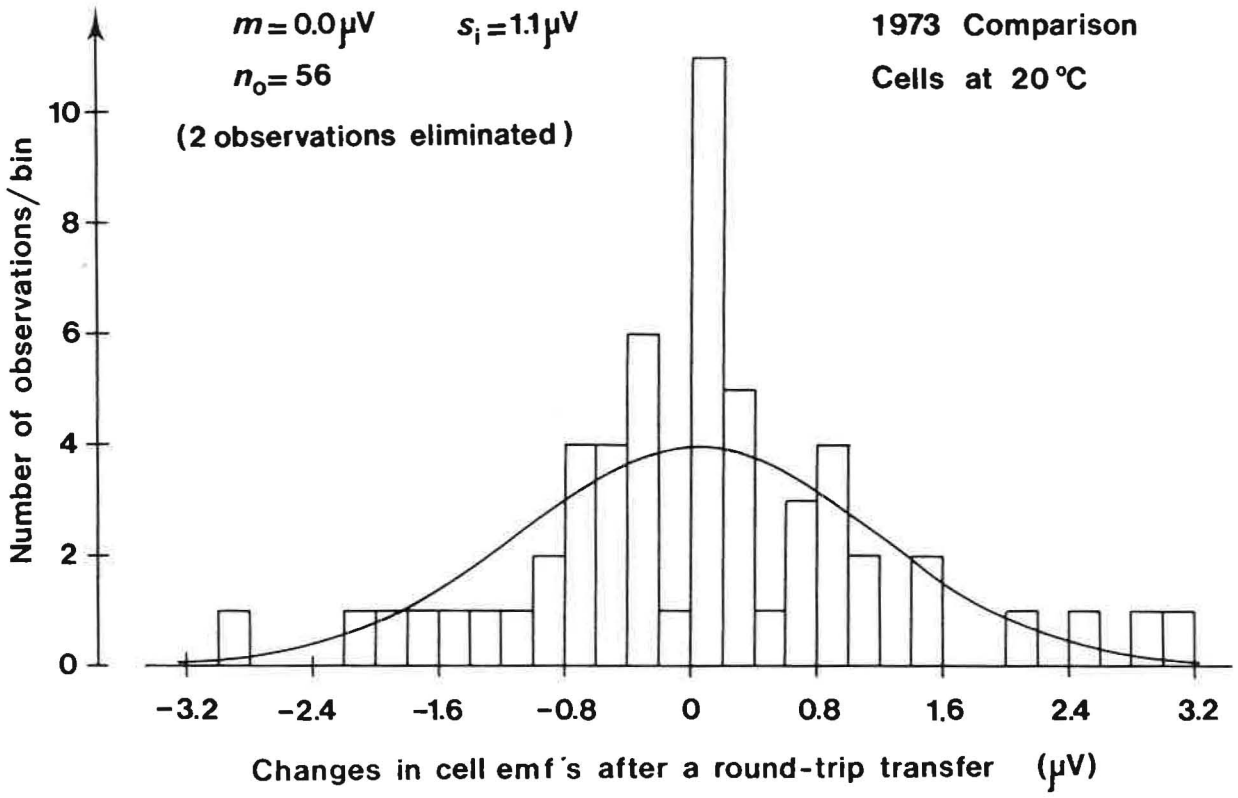

Fig. 12.- Changes in oil-bath cells at 20 °C for cells participating in the 1973 comparison. Two "outliers" were eliminated from the data.

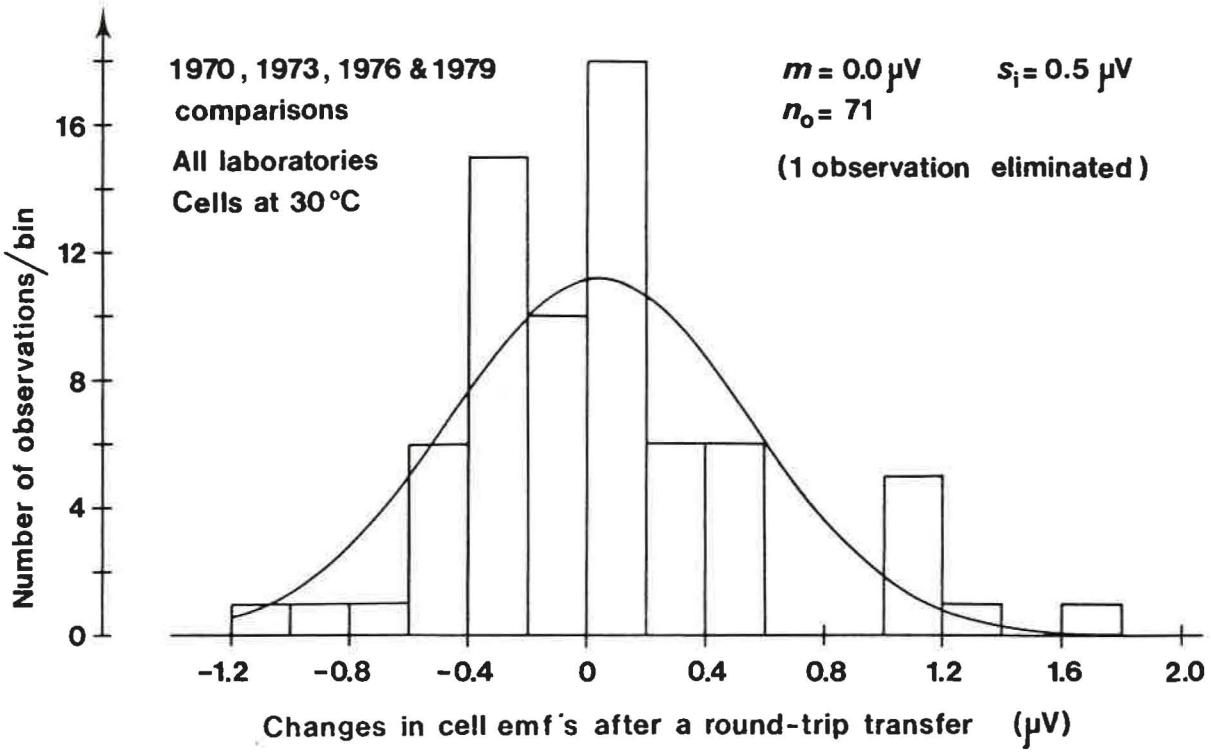

Fig. 13.- Changes in air-enclosure cells participating in the 1979, 1973, 1976 and 1979 comparisons. One "outlier" was eliminated.

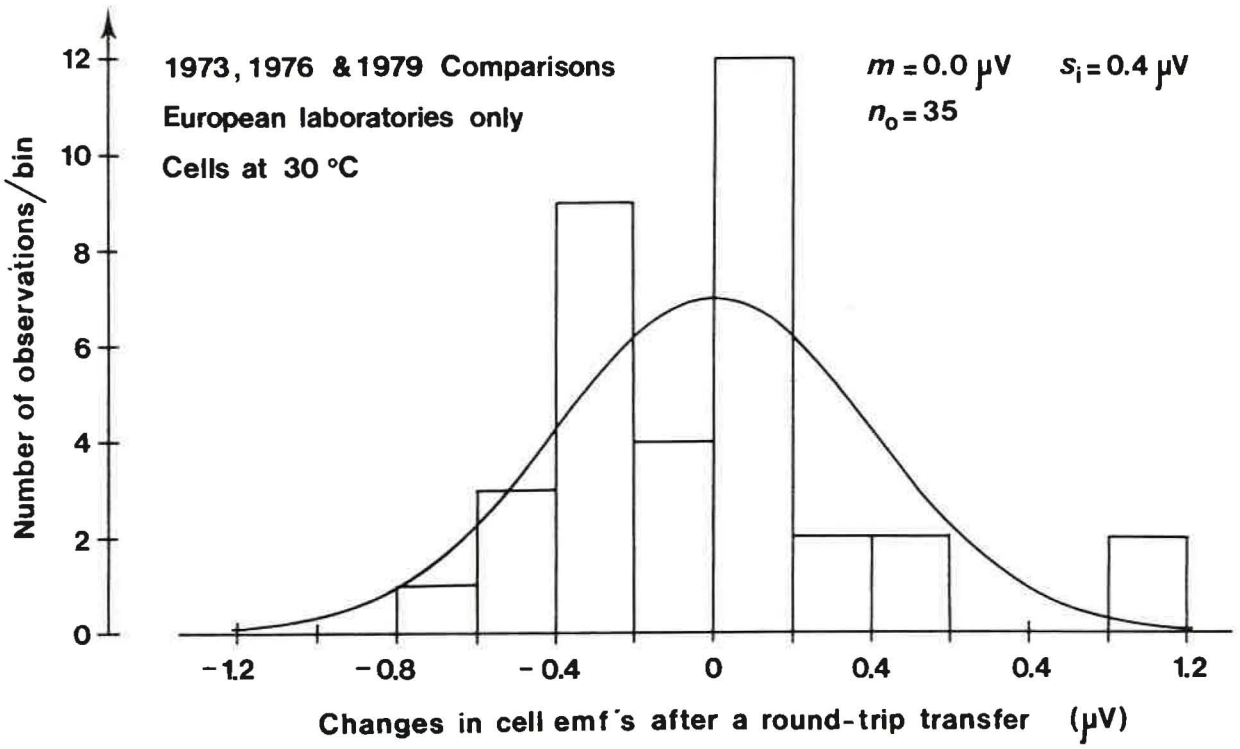

Fig. 14.- Changes in air-enclosure cells from European laboratories participating in the 1973, 1976 and 1979 comparisons.

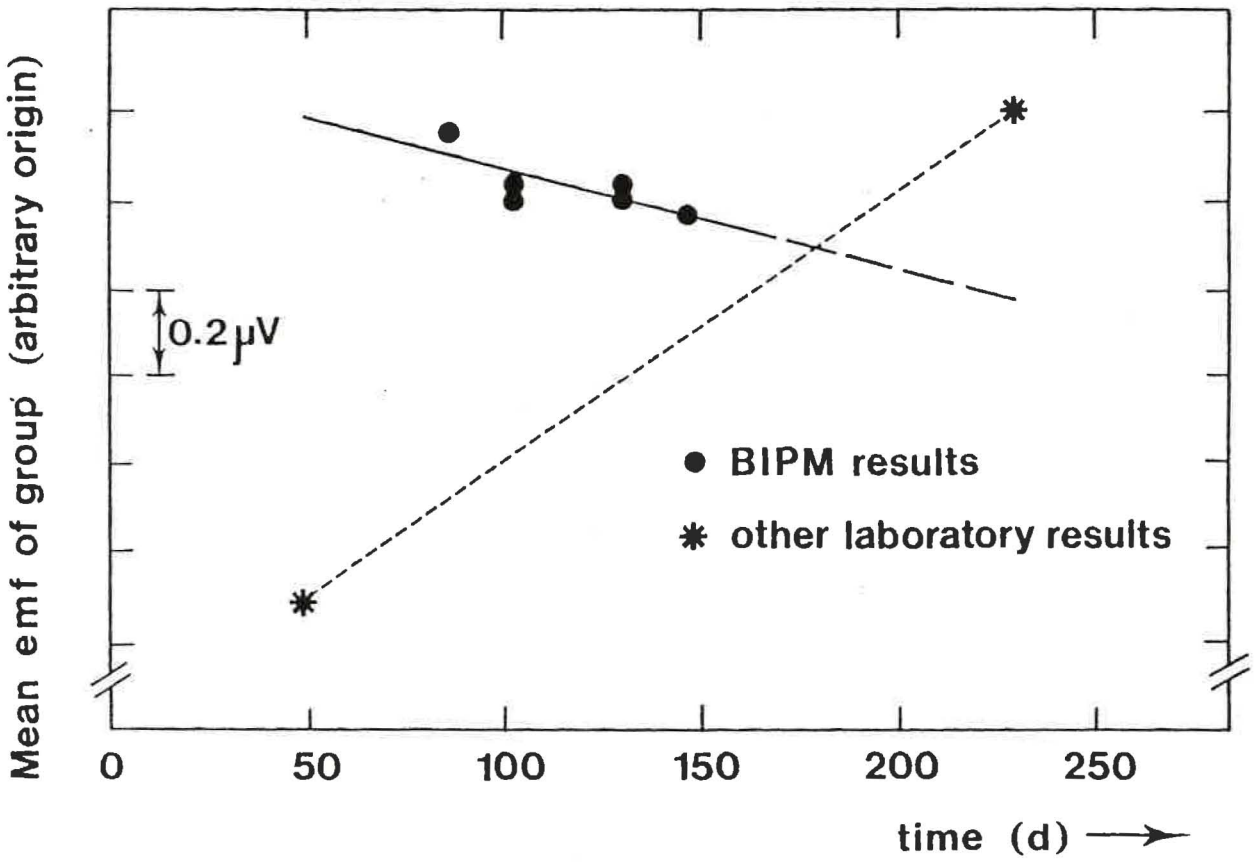

Fig. 15.- Values of the mean emf of a group of cells measured by the BIPM and by the laboratory originating the comparison. This illustrates the difficulties of extrapolating cell emf's after transporting the cells.

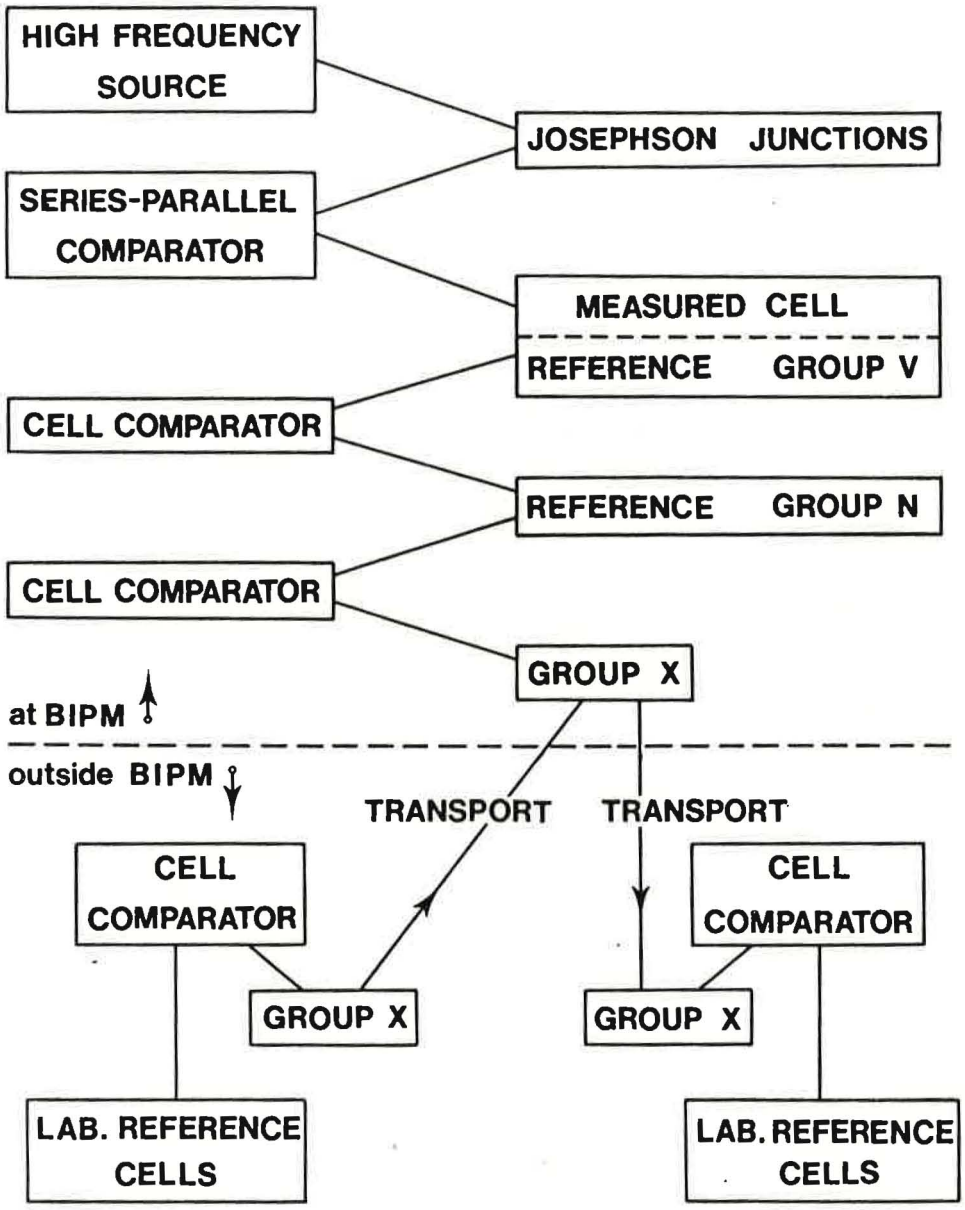

Fig. 16.- Schematic diagram of the principal steps and sources of uncertainty in the comparison of units of emf with an external laboratory by transport of standard cells.

Table I

Estimated Uncertainties* in the Determination of the Reference Cells in Terms of $V_{76\text{-BI}}$.

1. Uncertainties in the Josephson-effect measurement of one cell of group V			
series-parallel comparator, total		17	nV
random uncertainty of the mean		7	nV
thermal emf's		7	nV
frequency purity and measurement		10	nV
standard cell-related uncertainties, including :			
thermal emf's	4 nV		
leakage resistance	7 nV		
temperature stability	2 nV		
total	8 nV	8	nV
total		23.5	nV
2. Uncertainties in the comparison of the cell of group V, measured by the Josephson effect, with the cells of groups V and N			
random uncertainty	3 nV		
leakage resistance	1 nV		
thermal emf's	2 nV		
temperature stability, group N	1 nV		
temperature stability, group V	1 nV		
standard cell comparator	1 nV		
total	4 nV		
total uncertainty in BIPM reference cells		24	nV

* Uncertainties are expressed as one-standard-deviation estimates.

Table II

A Typical Standard Cell Comparison Design

Cell from group N	Cell from group V	Measured emf including thermal emf's	Cell from group N	Cell from group V	measured emf including thermal emf's
1	2	$N_1 - V_2 + e_1$	4	5	$N_4 - V_5 + e_7$
		$V_2 - N_1 + e_1$			$V_5 - N_4 + e_7$
2	2	$V_2 - N_2 + e_2$	5	5	$V_5 - N_5 + e_8$
		$N_2 - V_2 + e_2$			$N_5 - V_5 + e_8$
2	3	$N_2 - V_3 + e_3$	5	6	$N_5 - V_6 + e_9$
		$V_3 - N_2 + e_3$			$V_6 - N_5 + e_9$
3	3	$V_3 - N_3 + e_4$	2	6	$V_6 - N_2 + e_{10}$
		$N_3 - V_3 + e_4$			$N_2 - V_6 + e_{10}$
3	4	$N_3 - V_4 + e_5$	2	1	$N_2 - V_1 + e_{11}$
		$V_4 - N_3 + e_5$			$V_1 - N_2 + e_{11}$
4	4	$V_4 - N_4 + e_6$	1	1	$V_1 - N_1 + e_{12}$
		$N_4 - V_4 + e_6$			$N_1 - V_1 + e_{12}$

Table III

Estimated Uncertainties* in the International Comparison of Units of emf by the Transport of Standard Cells to the BIPM

1. Uncertainty in BIPM reference cells (Table I)		24 nV
2. Uncertainties in the comparison of good-quality saturated cells at 30 °C at BIPM		
a. Standard deviation of the mean	8 nV	
b. Leakage resistance	2 nV	
c. Temperature stability	3 nV	
d. Thermal emf's	2 nV	
e. Standard cell comparator	1 nV	
total	9 nV	9 nV
3. Uncertainty in extrapolation of emf drifts of cells		50 nV
4. Uncertainty in comparison of cells at the other laboratory (assumed same as 2)		9 nV
5. Uncertainty in stability of cells due to transport		500 nV

* Uncertainties are expressed as one-standard-deviation estimates.