EVOLUTIONS IN THE DEVELOPMENT OF A MEASUREMENT REFERENCE STANDARD FOR THE TEMPERATURE RANGE ABOVE 962 ºC

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Lucrarea prezintă activităţile de realizare de artefacte şi măsurări efectuate la Institutul Naţional de Metrologie în scopul etalonării termometrului de radiaţie LP3 din dotare la punctul fix de definiţie a SIT-90 de solidificare a cuprului, ca etapă iniţială pentru realizarea SIT-90 în domeniul temperaturilor înalte situate peste 962 °C. Totodată sunt prezentate consideraţii generale asupra etaloanelor şi a metodelor de lucru la materializarea şi menţinerea SIT-90 în domeniul termometriei de radiaţie.

The paper describes the development of artifacts and performance of measurements in the National Institute of Metrology with the purpose of calibrating its own radiation thermometer LP3 at the ITS-90 defining fixed-point of solidification for copper, as an initial stage in the realization of ITS-90 in high temperature range above 962 ºC. It also makes general considerations on the measurement standards and working methods used for the materialization and maintenance of ITS-90 in radiation thermometry.

Keywords: The International Temperature Scale of 1990, radiation thermometer, blackbody radiation, fixed point calibration

1 Introduction

The progress of the National Institute of Metrology (NMI) in the practical realization of the ITS-90 (International Temperature Scale of 1990) [1] in the range of high temperatures above 962 ºC is presented in this paper. The starting point in the achievement of this purpose is the use of a photoelectric monochromatic radiation thermometer owned by the NMI - Thermometry laboratory, one of those currently used by the national metrology institutes at the top of the traceability scheme of temperature measurements results.

The ITS-90 materialization at these high temperatures, involves in the first place the development and utilization of a fixed point blackbody as a primary reference standard for the calibration of the radiation thermometer, as indicated in Chapter 2. The works performed in the NMI laboratory are presented in Chapter 3 and Chapter 4.

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2 General considerations on the measurement standards and working methods used for the materialization and maintenance of ITS-90 in radiation thermometry

2.1 General

The measurement of a body temperature by means of a radiation thermometer is based on the temperature dependence of spectral radiance from that body. In the particular case of a *blackbody*, this dependence is established by the Planck relation (1).

$$L_{\lambda,bb}(T) = \frac{c_1}{\pi \cdot \lambda^5 \cdot [\exp(c_2/(\lambda \cdot T)) - 1]} \tag{1}$$

where $L_{\lambda,bb}(T)$ is the spectral radiance, for non-polarized radiation of wavelength in vacuum λ of the blackbody, at the T thermodynamic temperature, while c_1 and c_2 are the first constant and the second constant of the radiation (c_1 = 3,741 774 9×10^{-16} W·m^2; c_2 = 1,438 769×10^{-2} m·K).

In the range of high temperatures above the Ag solidification point of 961,78 ºC, the practical temperature which approximates the thermodynamic temperature [2, 3], that is the *International Kelvin Temperature* T_{90}, is defined by ITS-90 in the relation

$$\frac{L_{\lambda,bb}(T_{90})}{L_{\lambda,bb}[T_{90}(X)]} = \frac{\exp\left(c_2[\lambda \cdot T_{90}(X)]^{-1}\right)\text{-}1}{\exp\left(c_2[\lambda \cdot T_{90}]^{-1}\right)\text{-}1} \tag{2}$$

where $T_{90}(X)$ is one of the following temperatures specific to the solidification points (defining fixed points): T_{90}(Ag) = 961,78 °C, T_{90}(Au) = 1064,18 °C şi T_{90}(Cu) = 1084,62 °C, while $L_{\lambda,bb}(T_{90})$ and $L_{\lambda,bb}[T_{90}(X)]$ is the spectral radiance in the relation (1), at the temperature T_{90} and $T_{90}(X)$.

2.2 Realization and maintenance of ITS-90

The equation (2) involves that the instrument used, that is the radiation thermometer, is of monochromatic type with a known effective wavelength and that at least the reference source at the calibration temperature $T_{90}(X)$ is the blackbody immersed in the solidifying melted metal (silver, gold or copper) [4].

Irrespective of the solution chosen for the practical realization of ITS-90, the *fixed point calibration* is required as a main operation which allows the determination of the output measurement signal of the radiation thermometer proportional with $T_{90}(X)$ of the equation (2).

The national metrology institutes generally use the following three solutions of practical realization of ITS-90 above 962 ºC [5]:

(i) The result of the fixed point calibration is transferred to a reference wolfram strip lamp, determining the current at the lamp which corresponds to a radiance temperature of the lamp equal to the fixed point temperature.

The different temperatures T_{90} are established and then maintained on the lamp by measuring the radiance ratios and obtaining a series of pairs of current values and radiance temperature. The radiance ratios are to be adjusted, when necessary, depending on the non-linearity of the thermometer. An interpolation relation between temperature and current may be established to provide a continuous connection between the two quantities.

(ii) The result of the fixed point calibration is transferred to a reference wolfram strip lamp.

The different temperatures T_{90} of a source are established by measuring the output signal related to the source at T_{90} and the signal related to the reference lamp at fixed point temperature, and measuring the ratios between the two signals using the definition equation of ITS-90. The signal ratios are adjusted, when necessary, depending on the non-linearity of the thermometer.

(iii) The result of the fixed point calibration is maintained on the thermometer.

The different temperatures T_{90} are established as a function of the output signal values of the thermometer. The signal ratios are calculated with reference to the output signal representative for $T_{90}(X)$ and, if necessary, adjusted for the non-linearity of the thermometer.

3. Measurement standards used

3.1 Radiation thermometer

The radiation thermometer used to perform the measurements described in this paper is a photoelectric monochromatic type LP3, with series number 80-36, designed and manufactured by IKE (University of Stuttgart) employing the measurement principle developed by PTB. This principle is based on the photoelectric effect produced by a thermal radiation flux with a narrow wavelength band, incident on a detector. Its output signal is proportional to the radiance of the incident radiant flux. This is why the radiation thermometer LP3 is also called "linear".

Figure 1 presents the main constructive elements of the radiation thermometer LP3. The thermometer LP3 consists mainly of the optical system, photodiode (silicon thermal radiation detector) and the measurement signal evaluation system. The frontal lenses making up the objective 1 form an image of the radiation source (the source is not present in the figure) in the plan of the *field diaphragm* 2. The field diaphragm consists of a circular aperture with a diameter of 0,25 mm made in a plane mirror named "field stop". The reflected image may be observed by the human operator through the ocular 3 of an adjustable telescope allowing the proper focus and alignment of the optical system. The field diaphragm's aperture defines the measuring area, which is the area of that part in the source surface which sends the radiation to be measured by the thermometer. The radiations crossing the field diaphragm are converted into parallel rays on passing through a collimating lens. They go further through one of the neutral filters 4 (filter A), the *opening diaphragm* 6 whose circular aperture has a diameter of 10,5 mm and which allows only the passage of those rays leaving the source at a solid angle, and one of the interferential spectral filters 5 (filter B). Then the radiation is focused on the selected silicon detector type Hamamatsu S1336-18BK 7, by means of an optical adapter consisting of a three-lens adjustment system. The filters are fixed in two rotating holders A and B allowing them to be successively used. The protection cell 9 having a controlled and constant temperature, incorporates both the photodiode and the electronic circuit processing the photocurrent 8 which includes the analog/digital converter (ADC). The numerical display device 10 located on the frontal panel, displays simultaneously the photocurrent and radiance temperature and alternatively, the temperature of the thermostated protection cell which is pre-set at 29,3 ^{o}C, and the filter selectors, switches 11 and 12. The frontal panel also features the power connecter and coupling connector to the calculator by interface RS 232. For the purchase and processing of the measured values, the program LP3DE provided by the producer is used.

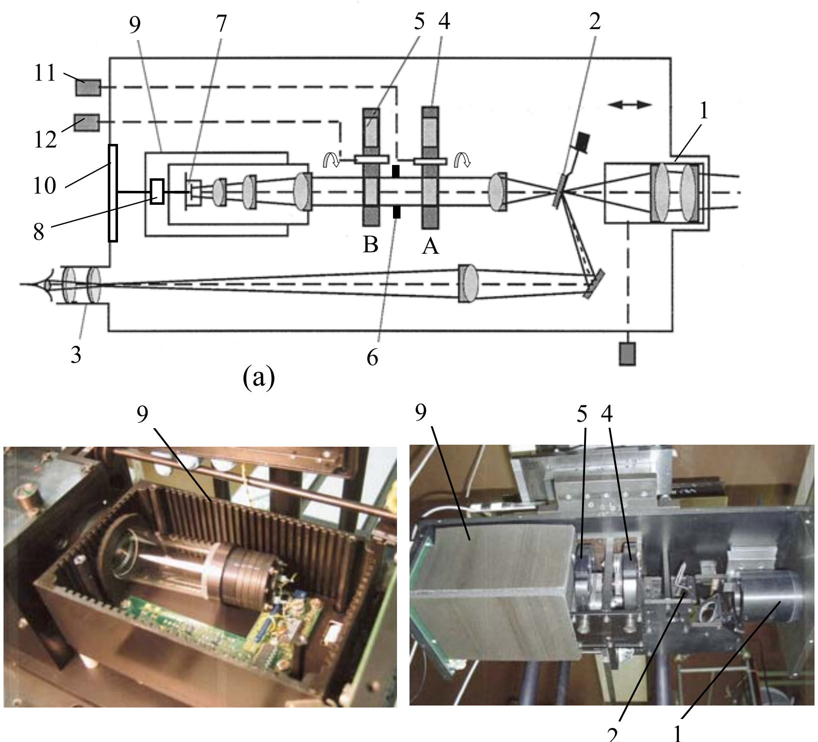
As it features the possibility to change the objective lenses, the spectral filters, the neutral filters and field diaphragm, the radiation thermometer LP3 may be adapted to a variety of sources to be measured. The nominal value of the wave length defined by the spectral filter, given by PTB, is λ= 651,9 nm corresponding to position B1 of the selector, where the spectral band width is of 10 nm HBW.

The nominal values of the transmission factor of the grey glass (neutral filter) are: τ = 1,0 and τ = 0,01 corresponding to the positions of the selector A1 (without a grey glass) and of selector A2. The diameter of the target source whose temperature is to be measured belongs to the range 0,6 mm … 1,8 mm which corresponds to the range 400 mm … 1000 mm of the distance between the target and frontal lens. The measurement ranges given by the constructor are: for temperature, from 950 K to 2600 K with the filters A1B1 and to 3800 K with the filters A2B1, while for the output signal (photocurrent), from 50 pA to 800 nA, with the possibility of selecting, irrespective of the filter combinations, one of the sub-ranges: R1 (50 pA … 8 nA) and R2 (1 nA … 800 nA).

Considering that the ratio in the relation (2) may be determined by the measurement of the photocurrents when the thermometer is successively compared with the reference source at temperature T_r, and the source to be measured at the unknown temperature T_x, the average effective wavelength λ_{ef} which depends on the two temperatures and the effective wavelength limit λ_{efl} [6, 7], approximation of the Planck relation (1) to the Wien relation [6, 8], then the calculation relation of temperature T_x in terms of the photocurrent i_{fc} displayed by thermometer LP3, is [9]:

$$T_x = \left[\frac{1}{T_r} - \frac{\lambda_{ef}(T_x, T_r)}{c_2} \cdot \ln \frac{i_{fc}(T_x)}{\xi \cdot i_{fc}(T_r)} \right]^{-1} \quad (3)$$

T_r and $i_{fc}(T_r)$ are determined from calibration or are taken from specifications [9]. ξ is a correction factor which takes into account the spectral emissivity of source, the transmittance of the windows and neutral filters and the reflectance of mirrors, as well as other optic components which are present in the optical path, but were not present and taken into consideration at calibration.



(a)

(b) Fig. 1 (c)

A silicon photodiode type detector is known for its excellent stability and linearity properties. That is why the silicon photodiode is recommended to be used in radiation thermometers which operate directly with the photocurrent and where the temperature-signal relation is based on the radiation theory. Such a thermometer, like LP3, is used as a standard for the maintenance and transfer ITS-90 in compliance with scheme (iii) at point 2.2.

The producer provides also for another relation, more effective, (4), used by thermometer LP3 to calculate the temperature T_c of the measured source in terms of the photocurrent i_{fc} [10, 11, 12] which is not based on the ration relation (2) and allows the calculation of the effective wavelength limit [6, 7].

$$T_c = -\frac{c_2}{A \cdot \ln\left(\dfrac{i_{fc}}{\xi \cdot C}\right)} - \frac{B}{A} \tag{4}$$

where A, B and C are constant parameters, specific to each thermometer, which are determined usually by calibration of the thermometer at no less than three reference temperatures. Another way to determine the three parameters is given in [13].

For the thermometer LP3, the constants A, B, and C were determined and given by the producer, but they may be reconfigured when necessary, without the producer's contribution.

The relation (4) determines the effective wavelength limit which is calculated by the empirical relation (5) [10, 11] and is used to determine the average wavelength from (3). However, PTB has approximated this wavelength with the value λ_{ef} = 651,9 nm. The differences between T_c and T_x found by PTB for the thermometer LP3 in the range temperature (1050 … 2300) K are within -0,72 K to 0,31 K.

$$\lambda_{efl}(T_c) = \frac{(A \cdot T_c + B)^2}{A \cdot T_c^{\,2}} \tag{5}$$

3.2 The blackbody and the oven used to realize and reproduce the solidification point for copper (1084,62 ºC)

According to Kirchoff law [14], the thermal radiation emitted by the walls inside a closed cavity which are in a thermodynamic equilibrium is a black body radiation. The spectral density of a volumetrical radiant energy $w_\lambda(T)$ is the same at any point inside the cavity, depends on the wavelength λ and the temperature T, but does not depend on the material of which the walls are made or on their shape. Access into this cavity is made through a small hole in the cavity walls, like the hole 1 in figure 2. The radiation emitted through the small hole is considered only the radiation generated inside the cavity, which is a good approximation of the black body radiation. The surface area of the small hole in the wall behaves like a black body. The smaller the surface area of small hole compared to the surface of inner walls, the better the black body approximation through the surface area of the small hole.

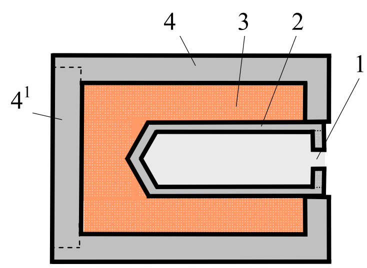


Fig. 2

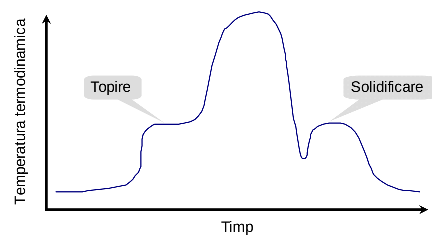


Fig. 3

Figure 2 shows the schematic design of a black body designed and achieved in the NMI laboratory, while figure 4 shows two images of the crucible containing the black body. The construction consists of three parts: black body 2 provided with a diaphragm delimiting the circular sighting hole 1, the crucible 4 also containing the lid 4^1 and the metal (copper) 3 where, when melted, the blackbody is immersed, reaching the fixed point temperature ITS-90 for copper solidification. The typical curve describing the melting and solidification of the metal is presented in fig. 3.

Fig. 4

The blackbody has a sighting length of 80 mm, the inner diameter of 8 mm and wall thickness of 1 mm. The sighting hole of the radiant cavity where the thermodynamic equilibrium at the copper solidification temperature is achieved has 1 mm in diameter. The cylindrical body of the crucible is 120 mm long, 30 mm in outer diameter and 4 mm in wall thickness. The black body is tightly fixed in the crucible by pressing.

The cylinder body and the closing lid which are also tightly fixed by pressing will have an aperture of 0,5 mm to allow the overflowing copper to be evacuated in case of overheating. All the components are made of pure spectral graphite with impurity content lower than 5×10^{-6}. The choice of graphite is dictated by its excellent qualities of emissivity, thermal conductivity and workability.

For filling with copper and the subsequent reproduction of solidification point, the black body is introduced in an oven like that in Fig. 5, mainly consisted of: casing 1, lids 2 and 3, crucible 4, copper 5, black body 6, sighting cone 7, press disks 8 and 9, aluminum oxide tube 10 with the heating resistor 11, aluminum oxide diaphragms 12 and 13, ceramic fiber insulation 14 and 15. At the back side of the oven there is a connecting piece 16 for argon filling and a hole 17 for temperature control thermocouple.

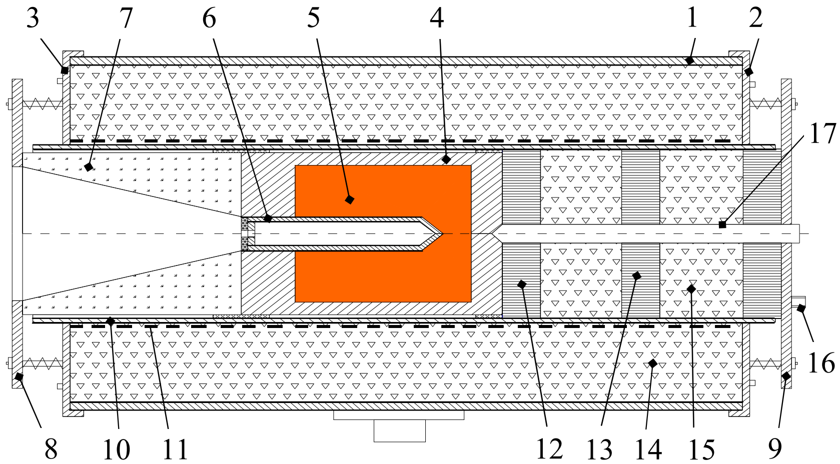


Fig. 5

The images in fig. 6 are views of the two frontal faces of the oven: 6 (a) is one with the sighting cone.

(a) (b)

Fig. 6

The copper filling is made with the oven in a vertical position while the whole assembly is maintained at a temperature with (3…5) ºC higher than the copper melting temperature. The operation was performed in argon atmosphere. After filling the crucible with copper, the lid was tightly pressed.

The hole of the black body is delimited by a platinum diaphragm which facilitates the thermometer centering. Because the platinum emissivity is much lower than the graphite emissivity, the hole of the black body appears much brighter than the platinum ring at high temperatures.

The copper quantity for filling the crucible is about 800 g and its purity should be higher than 99.99 %.

The calculated spectral emissivity ε_λ of the black body cavity is higher than 0.9999. In conclusion, the luminance temperature of the black body is the same with the real temperature while the corrections to be made are considered null.

4. The calibration of the radiation thermometer LP3 to the blackbody for copper solidification point (1084,62 °C)

The black body of copper fixed point (solidification point) achieved by the NMI and presented at point 3.2 was used for the first time in 2007 for the calibration of the radiation thermometer LP3. At that stage, the calibration mainly consisted in comparing the thermometer indications to the reference black body.

The thermometer LP3 had been previously calibrated at PTB (Germany) by comparison with two wolfram strip temperature lamps type Polaron, one with high vacuum and series P95 and the other with pressurized gas and series P98. A primary calibration was not performed at PTB, which involved calibration at black body. In this case, the gold point value was preserved on the temperature lamp P95. The calibration certificate issued by PTB establishes the relation between the temperature indicated by the thermometer LP3 and the spectral radiance temperature of the standard used for the calibration of the thermometer (wolfram strip lamp). The extended uncertainty obtained at PTB for spectral radiance temperatures between 1050 K and 2300 K, falls in the range (0.7…2.3)K depending on the temperature values.

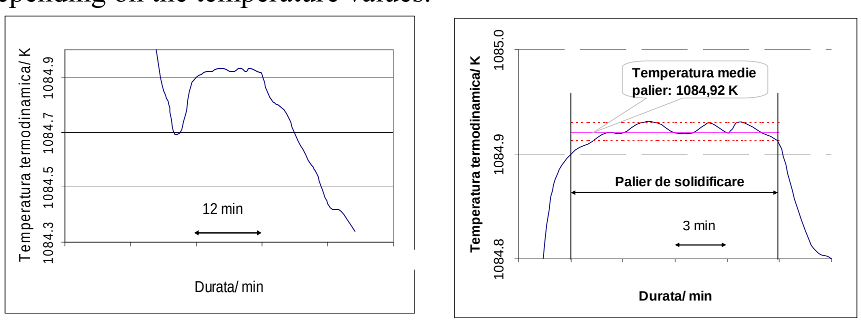


Fig. 7

For the calibration of the thermometer LP3 at copper fixed point black body at NMI, the distance between the pyrometer objective and the black body hole was of 630 mm. The power supply of the oven through the autotransformer was of 7.5 A for melting and between 6,2 A and
6,5 A for solidification. Five solidification plateaux with durations ranging of (13…21) minutes were performed within two weeks. The measurement results were registered every minute and displayed and stored on a computer. For each plateau, the average of the values indicated by the thermometer LP3 was calculated and the deviations of the averages were found within the limits of

(+0.01…+0.30) ºC. The average of the five plateaux was t_{CuI} = 1084.80 ºC, different by 0,18 ºC from the ITS – 90 (t_{Cu} = 1084,62 ºC). Fig. 7 shows the graphics of one of the above-mentioned solidification plateaux. According to the [9], because the deviation of the average value keeps under 0,2 K, the values of the previous calibration remain valid [15].

5 Conclusion

The fixed point calibration is to be concluded by completing the measurements presented in Chapter 4 with the stage of uncertainty sources evaluation and of measurement uncertainty estimation.

The standards and measurements presented in Chapters 3 and 4 provide a good premise for the NMI to continue the research and the ITS-90 materialization in radiation thermometry, mentioned in the three solutions of practical realization referred to at point 2.2.

R E F E R E N C E S

[1] *H. Preston-Thomas*, The International Temperature Scale of 1990 (ITS-90), Metrologia, 1990, **27**, 3-10
[2] *D. Dinu*., Considerații privind scările de temperatură, Metrologie, 2006, vol. LIII (serie nouă), nr. 1-4, 61-75
[3] *D. Dinu D., M.M. Poenaru*., Considerations on future redefinition of the kelvin*,* Scientific Bulletin, Series A: Applied Matematics and Physics, Editat de Politehnica Press, University Politehnica of Bucharest, 2009, Vol. **71**, Issue 2, 95-104
[4] xxx Suplimentary Information for the International Temperature Scale *of 1990,* Bureau International des Poids et Mesures (BIPM), Pavillon de Breteuil, F-92310 Sevres, 1990
[5] xxx Uncertainty budgets for realisation of scales by radiation thermometry, BIPM, CCT-WG5 on radiation thermometry, Document CCT/03-03, 2003
[6] *G. D. Nutter*, Radiation thermometers: design principles and operating characteristics, Theory and practice of radiation thermometry, John Wiley & Sons, Inc, New York, 1988, 231 – 337 (cap. 4)
[7] *J. Bezemer,* Spectral Sensivity Correction for Optical Standard Pyrometers, Metrologia, 1974, **10**, 47 - 52
[8] *J. Hartmann*., High temperature measurement techniques for application in photometry and thermometry, Physics Report, 2009, **469**, 205-269
[9] *G. Neuer, E. Schreiber*, Linearpyrometer LP3. Operating Instruction, Data Sheat Instrument No. 80-36, Forschungsinstitut Fur Kerntechnik und Energiewandlung KE E.V., 1999
[10] *F. Sakuma., S. Hattori*., Establishing a practical temperature standard by using a termometer with a silicon detector, Temperature: Its Measurement and Control in Science and Industry, Vol. 5, Editat de: J.F. Schooley, New York, American Institut of Physics, 1982, 421-427
[11] *F. Sakuma., M. Kobayashi*., Interpolation equation of the scales of radiation thermometers, Temperature: Its Measurement and Control in Science and Industry, Vol. 5, Editat de: J.F. Schooley, New York, American Institut of Physics, 1982, 305-310 (Publicat prima data in 1994, in japoneza)

[12] *A. Ono, S. Hattori, F. Sacuma*, Detector-based traceability of temperature standards, Theory and practice of radiation thermometry, John Wiley & Sons, Inc, New York, 1988, 839 – 857 (cap. 15)

[13] *F. Sacuma, M. Laina*, Calibration and caracterization of the Transfer Standard Radiation Thermometer for an APMP Intercomparison, Temperature: Its Measurement and Control in Science and Industry, Vol. 7, Editat de: D.C. Ripple, New York, American Institut of Physics, 2003, 589-594

[14] *I.M. Popescu*, Fizica termodinamică, Editura Politehnica Press, Bucureşti, 2002

[15] xxx Calibration certificate, CE 3927 PTB 99, 2000