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# Measurements of the Refractive Index of Air Using Interference Refractometers

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**Abstract**

Comparisons have been carried out between interference refractometers built in different countries. Individual measurements of the refractive index of air have been made using air from the same sample volume. Direct comparison of refractometers was realized by coupling the instruments to the same air-inlet system.

In order to compare the measured results the refractive index was also calculated from accurate measured values of pressure, temperature, humidity and $CO_2$ content, using Edlén's formula. Most of the individual measurements show an agreement within 1 part in $10^7$ while direct comparisons show an agreement within 5 parts in $10^8$. Over a period of five days an increase in $CO_2$ content of 400 ppm per day was measured in a 300 $m^3$ laboratory room. The results show the great importance of correcting calculated results for the $CO_2$ content of the air.

## 1. Introduction

Laser interferometers are used for the accurate determination of length and the calibration of precision length-measuring equipment. An absolute accuracy of 1 part in $10^7$ is increasingly being demanded from these interferometers and this can only be realized in the free atmosphere by correcting the wavelength of the laser radiation for variations in the refractive index of the air. For this reason the accurate determination of refractive index is of vital importance to national standards laboratories. Therefore, three years ago the Community Bureau of Reference, Commission of the European Communities, Brussels, initiated a project on the comparison of air-refractometers developed in different laboratories of EC countries. The aim of the project was to obtain a better understanding of the accuracy of these refractometers, at a level of 1 part in $10^7$. Additionally, since the Edlén air-refractive-index formula [1] is widely used in industry and standards laboratories, the second objective of the comparison was to attempt to verify the reliability of this formula.

For the project a comparison set-up was built at the Metrology Laboratory of Eindhoven University of Technology and the measurements were made there between 6th and 10th of June 1983. The following laboratories are participating in the project:
- National Physical Laboratory (NPL) England,
- Physikalisch-Technische Bundesanstalt (PTB) West Germany,
- Van Swinden Laboratory (VSL) and
- Metrology Laboratory Eindhoven University (THE), both of the Netherlands.

In this paper a survey of experiments and results is presented; a complete description has been given in the BCR report, Ref. [2].

## 2. Brief Description of the Refractometers Used in the Comparison

### 2.1. The NPL Refractometer

This refractometer [3] consists of a double-passed Jamin beam-splitter, an air sample cell and a single retro-reflector of the cat's-eye type. A diagram of this set-up is given in Fig. 1. The surfaces of the beam-splitter block are specially coated so that equal interfering intensities for both polarization directions are produced, which results in maximum fringe contrast. The interferometer does not need a stabilized laser and due to its "common path" optical configuration it is insensitive to path-length variations resulting from mechanical changes. The design of the sample cell is also shown in the diagram. The cell consists of inner and outer glass

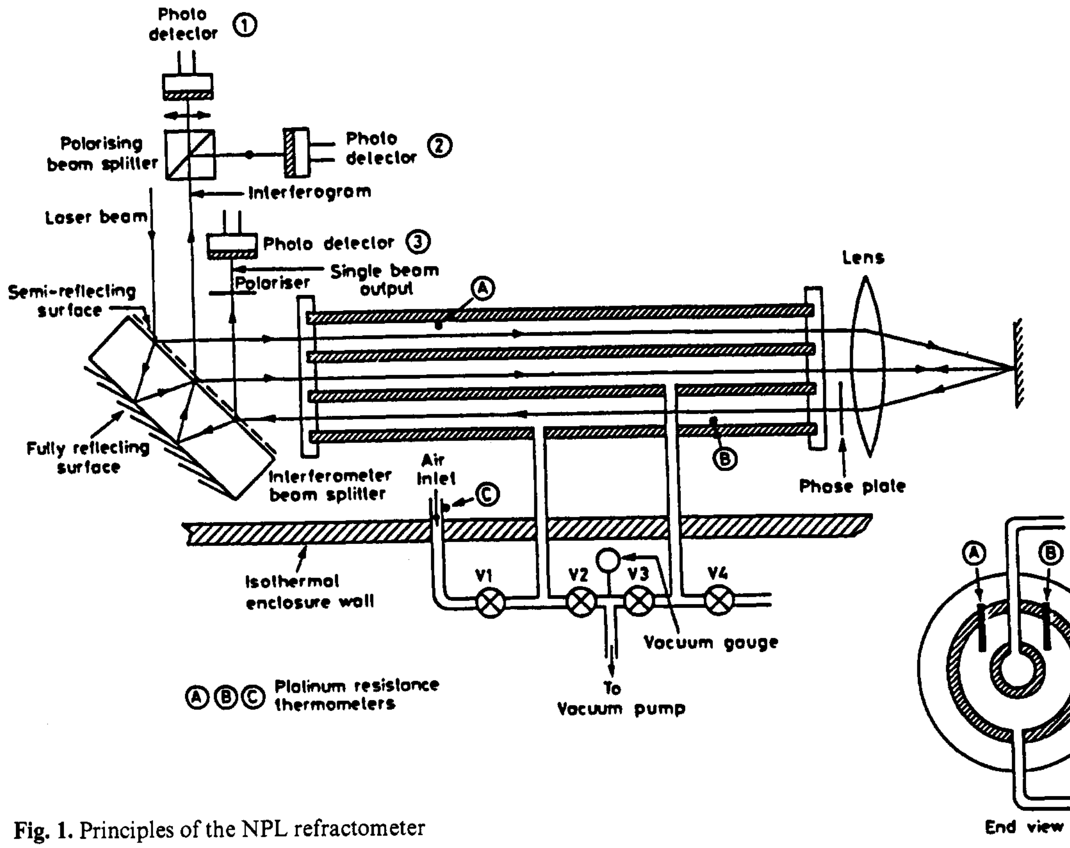


**Fig. 1.** Principles of the NPL refractometer

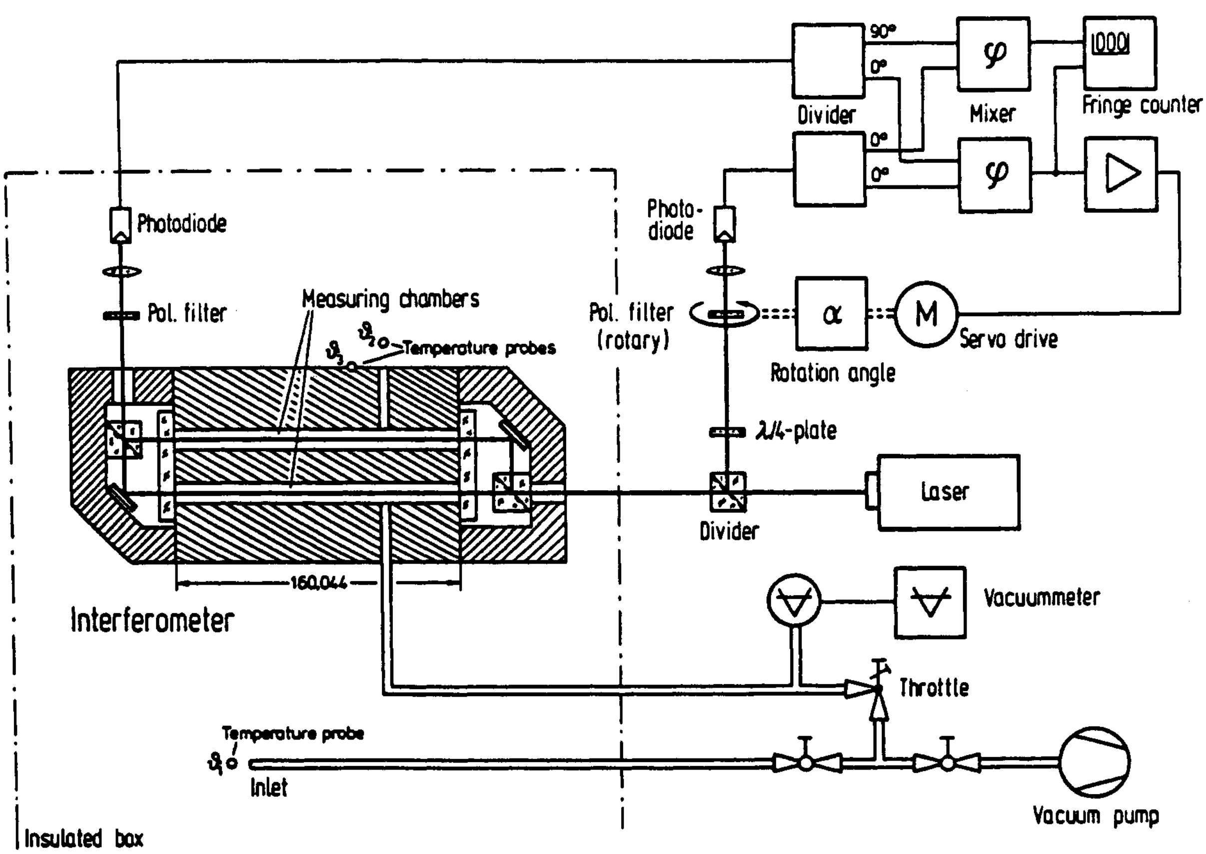


**Fig. 2.** The PTB refractometer

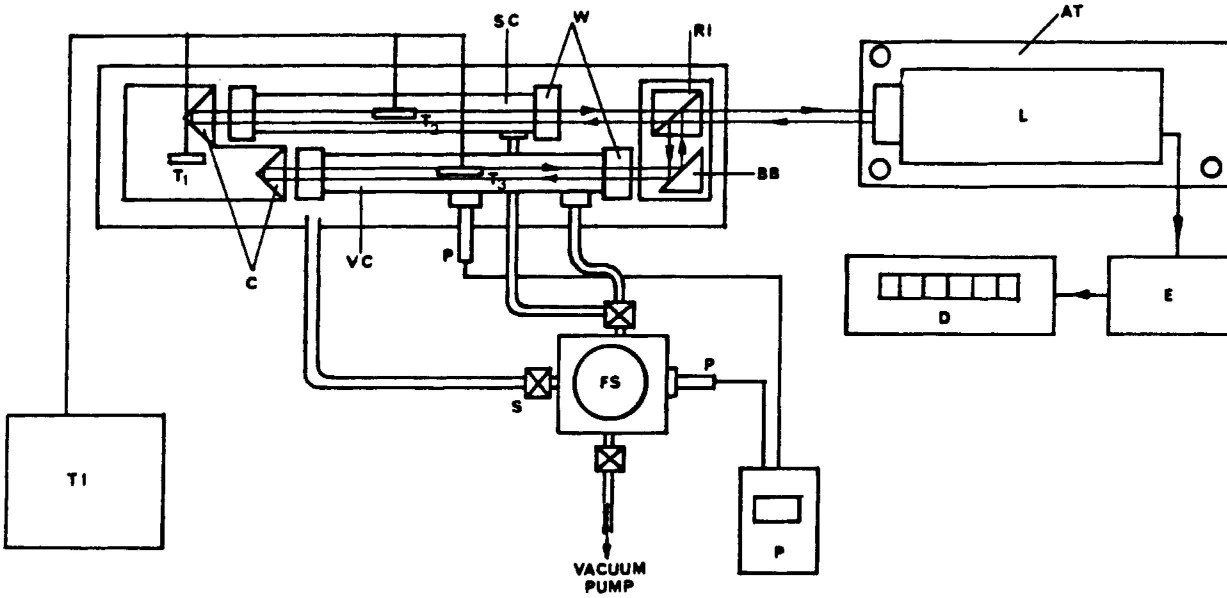


**Fig. 3.** Schematic of the THE refractometer. RI: remote interferometer; $T_1$, $T_2$, $T_3$: temperature measurement; L: laser; E: extender; D: display; C: cube corners; BB: beam-bender; SC/VC: sample cell/vacuum cell; FS: filling station; P: Pirani gauge; AT: adjustment table; S: sample valve; W: quartz windows; TI: temperature-measurement system

tubes of equal length with glass windows cemented on each end. Refractive index measurement is achieved by first evacuating the inner and outer cells and then air is admitted into the outer cell. With a bi-directional fringe-counting system the number of counts arising from this vacuum-to-air change is measured, while an interpolation can be carried out by measuring the angular orientation of the voltage vector composed from the signals of detector 1 and detector 2. Using this technique a resolution of 1 part in $10^8$ is achieved.

### 2.2. The PTB Refractometer

This refractometer [4] consists of a Mach-Zehnder interferometer using an integrated block with two bores and optical windows which serve as measuring chambers (Fig. 2). The interferometer is driven by a two-mode stabilized He-Ne laser with a beat frequency of around 600 MHz. Changes of the optical length of one of the arms of the interferometer produce phase shifts of the beat oscillation. These shifts are measured relative to the phase shifts of a reference path. Complete fringes (= 360° of phase change) are counted by a fringe counter whereas the high-resolving evaluation of the fringe patterns is done by tracing back phase shifts to the rotation of a polarizing filter. Thus, fractions of complete fringes are detected by measuring a rotation angle: 180° of rotation = 360° of phase = 1 complete fringe. The measurements were made first with both chambers filled with air as a reference. Then one chamber is pumped down and, allowing a certain time for settling of temperature, complete fringes and fringe-fractions are registered. From these values the refractive index has been calculated. The resolution of the interferometer is about 1 part in $10^8$.

### 2.3. The THE Refractometer

This instrument is based on an ordinary double-beam interferometer (Michelson type) using a commercially available laser-measurement system. A schematic diagram is shown in Fig. 3. The laser beams are passed through 400 mm long cells enclosed by quartz windows. The system uses the normal remote interferometer and cube corners; an extra beam-bending mirror is used so that both beams are parallel and close together. A 100 times electronic extension is used for fringe sub-division so that each count of the interferometer corresponds to $\lambda/400$ which gives a resolution of 5 parts in $10^9$ in the refractive index. Temperature inside the measuring or sample cell is measured by Pt-100 resistance thermometers. For the measurements of the refractive index both cells are first evacuated and then the air is admitted into the sample cell. After a short settling time the counter is read and the refractive index of the sample can be calculated from the number of counts and temperature data.

### 2.4. The VSL Refractometer

This refractometer is based on the same measurement system as the THE refractometer but the set-up and filling system are different. A schematic diagram is given in Fig. 4. The instrument can be

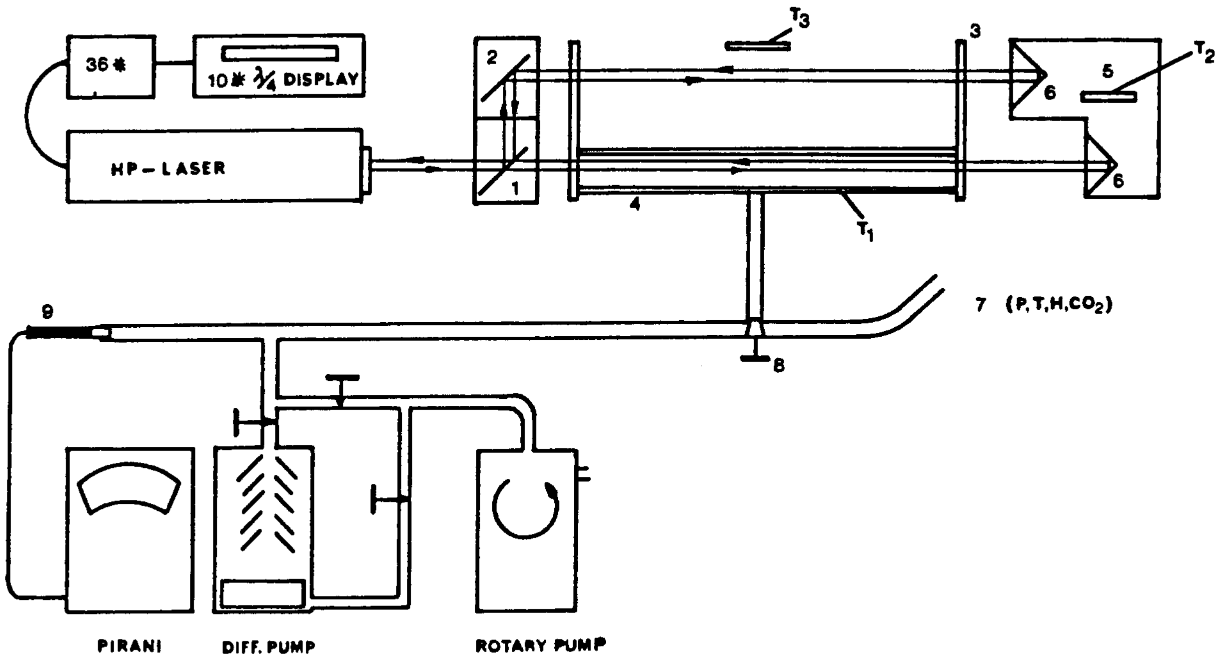


**Fig. 4.** Schematic of the VSL refractometer. 1. Beamsplitter; 2. beambender; 3. glass windows; 4. sample cell; 5. aluminium mounting block for cube corners; 6. cube corners; 7. air inlet; 8. triple valve for air inlet and pumping system; 9. pressure measurement; $T_1$: thermometer for sample cell; $T_2$: thermometer for the aluminium block; $T_3$: thermometer of the open-air path

seen as an inexpensive version of the THE refractometer and was assembled at short notice as one of the intended participants could not take part in the experiments. In this set-up the laser beams are also parallel but here one beam is passed through open air while the other beam passes through the sample cell. This cell is enclosed by glass plates extending into the air path of the interferometer. As with the THE refractometer the cube corners are staggered over about 50 mm to correct for asymmetry of the interferometer. A total extension of 360 times is used so that one count corresponds to $\lambda/1440$ but, because of an instability of a few counts, a resolution power of 5 parts in $10^9$ results for the sample-cell length of 280 mm. For the measurements of the refractive index the sample cell is first evacuated and then air is admitted slowly. After small corrections for changes in the open-air path, the refractive index is calculated from the number of counts resulting from the vacuum-to-air change in the sample cell.

## 3. The Comparison Set-Up

This set-up, shown schematically in Fig. 5, consisted of a large stone table carrying an insulated cabinet containing the four refractometers. The size of the stone table was $1.5 \times 3$ metre. The insulated cabinet, size $2.5 \times 0.75 \times 0.5$ metre, was constructed of 50 mm polystyrene covered on both sides by a layer of 5 mm pressed paper. Holes were drilled through the cabinet for feed-through of light beams and cables; the holes were closed by optical windows. The laser light sources as well as the vacuum pumps and other heat sources were situated outside the cabinet. One particular area in the cabinet was designated the sample place from which air was admitted into each of the refractometers. The sensors for the measurement of barometric pressure, air temperature and humidity were also mounted in this common area. The measurement of $CO_2$ content was carried out by taking air samples regularly during the measuring period and analysing them afterwards by gas chromatography. The temperature was measured by calibrated resistance thermometers (Pt-100, Pt-10) and semiconductor thermometers. The pressure measurements were made using both a barometer with quartz pressure sensor (Paro-Scientific) and a precision aneroid barometer (Negretti and Zambra). Humidity was measured by a dew-point-measuring instrument (humidity analyser 911 EG & G). Figure 6 shows the open cabinet containing the refractometers. With this set-up four kinds of experiments were carried out:

1) individual measurements of refractive index at the sample-place,
2) individual comparisons with Edlén's formula,

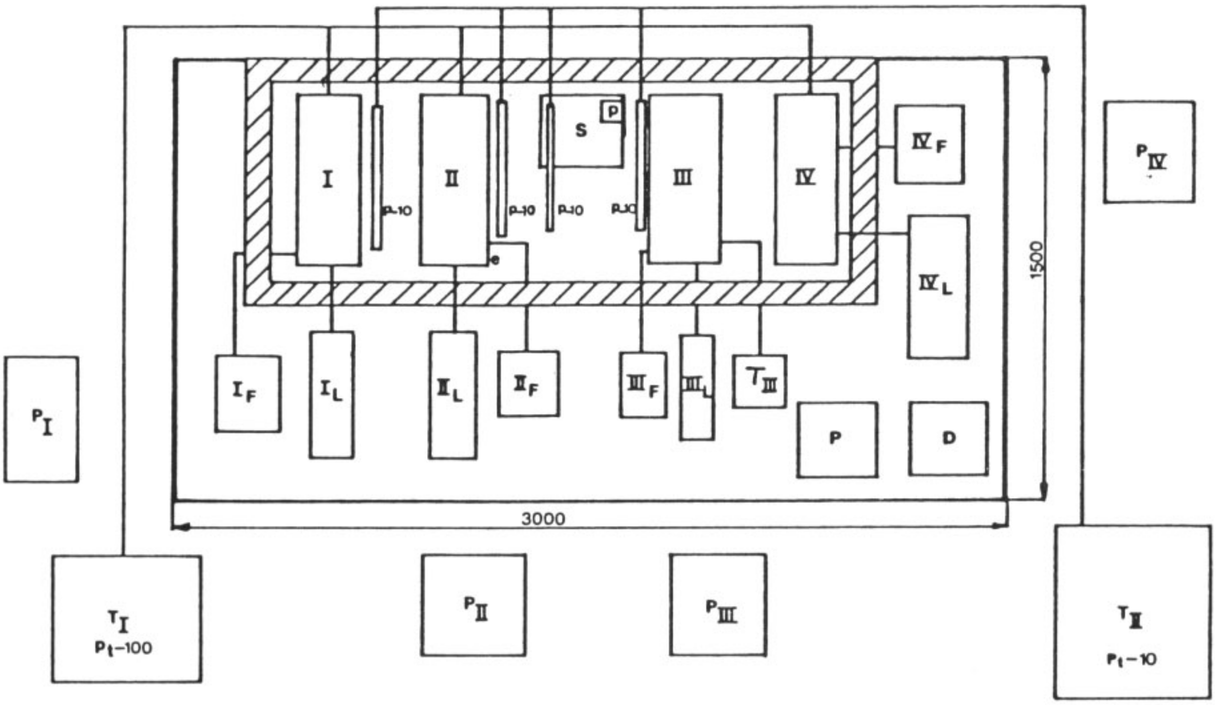


**Fig. 5.** Schematic diagram of comparison set-up. I ... IV: refractometers; $I_F \ldots IV_F$: air-inlet system; $I_L \ldots IV_L$: laser light source and counting system; $T_I \ldots T_{III}$: temperature-measurement system; $P_I \ldots P_{IV}$: pumping system; p-10: resistance thermometers; P: pressure measurement; D: humidity measurement; S: sample-place

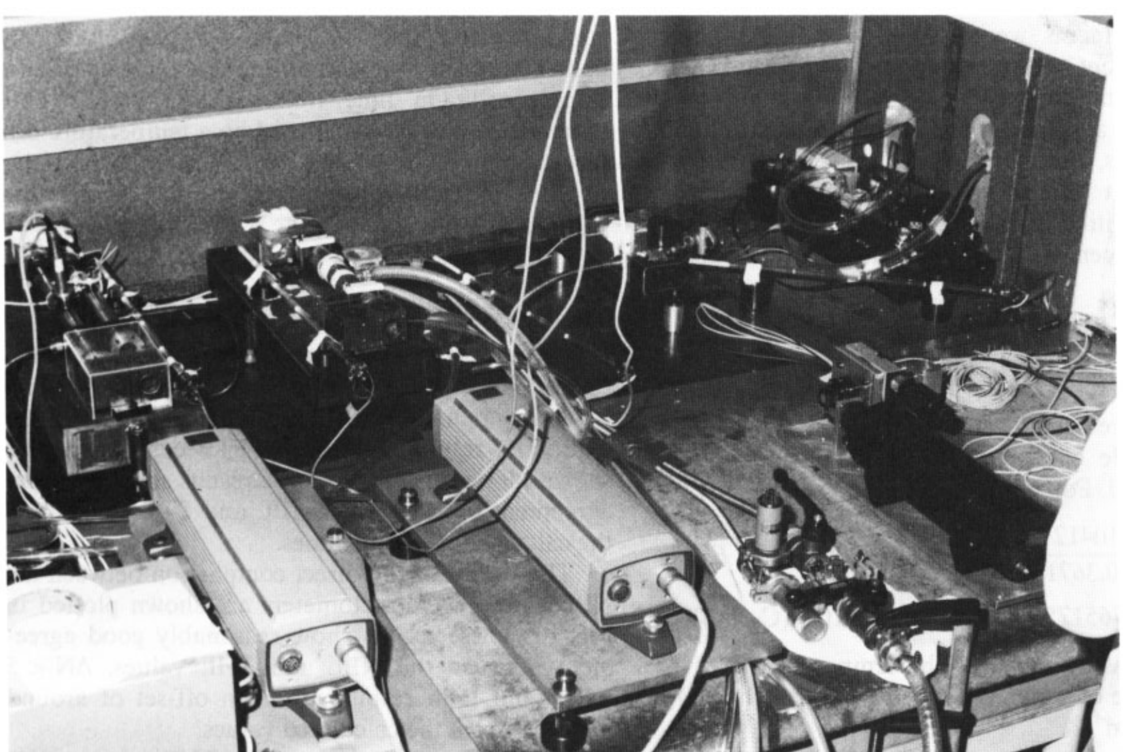

**Fig. 6.** Open cabinet with refractometers and laser systems

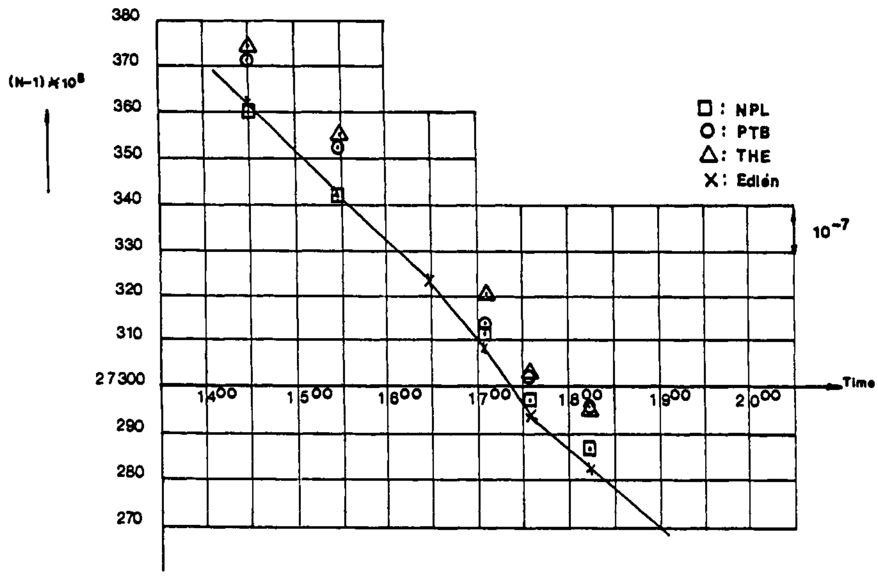


**Fig. 7.** Measurements of air refractive index on 7 June, 1983

3) direct comparison of refractometers by measuring the same air sample by coupling the inlet systems of two refractometers,
4) comparison of the difference between the measured refractive indices and values derived from Edlén's formula.

## 4. Results of the Measurements

The measurements were carried out immediately after adjustment of the refractometers and associated instrumentation over a period of four days between the 7th and the 10th of June, 1983. During the first two days, individual measurements of refractive index at the sample-place were performed whilst, in addition, for the last two days, direct comparisons between the refractometers were made also.

### *4.1. Results of Individual Measurements*

For these measurements the refractive index at the sample-place was also calculated from measurements of pressure, temperature, humidity and carbon-dioxide content using the following, somewhat modified, Edlén formula:

$$N - 1 = \frac{D\,(0.104127 \times 10^{-4})\,P}{1 + 0.3671 \times 10^{-2}\,T} - 0.42063 \times 10^{-9}\,F$$

with $D = 0.27651756 \times 10^{-6}\,[1 + 54 \times 10^{-8}\,(C\text{-}300)]$

($P$ is the pressure in Pa, $T$ the temperature in °C, $F$ the pressure of water vapour in Pa and C the $CO_2$ content in ppm [1]).

[1] ppm ≡ $1 \times 10^{-6}$

The results are plotted in Figs. 7 and 8.

The results in Fig. 7 do not include the VSL measurements since at that time this refractometer was not operative due to problems with the inlet system. The results in Fig. 8 show off-sets for the NPL results as compared to the calculated (Edlén) values. These were caused partially by the THE inlet system since, at that time, the NPL refractometer was connected to the THE inlet system which caused a mean off-set of $7.5 \times 10^{-8}$ over the measuring period due to changes in the humidity content during the air-filling procedure of the sample cell. On June 10th the VSL results were corrected for an off-set caused by a temperature difference between the outside of the sample cell (temperature-measuring place) and the air temperature inside this cell.

### *4.2. Results of Direct Comparison of Refractometers*

Direct comparison of refractometers was carried out on the 9th of June between the PTB and NPL refractometers and between the NPL and THE on the 10th. The results of the NPL-PTB comparison are shown plotted in Fig. 9. These results show excellent agreement both between NPL and PTB results and the calculated (Edlén) values.

The results of the direct comparison between the NPL and THE refractometers are shown plotted in Fig. 10. These results show reasonably good agreement between the NPL and THE values, $\Delta N < 5 \times 10^{-8}$, but both results suffer an off-set of around $7.5 \times 10^{-8}$ from the calculated values.

This off-set was due to the THE inlet system which was used by both refractometers. This caused

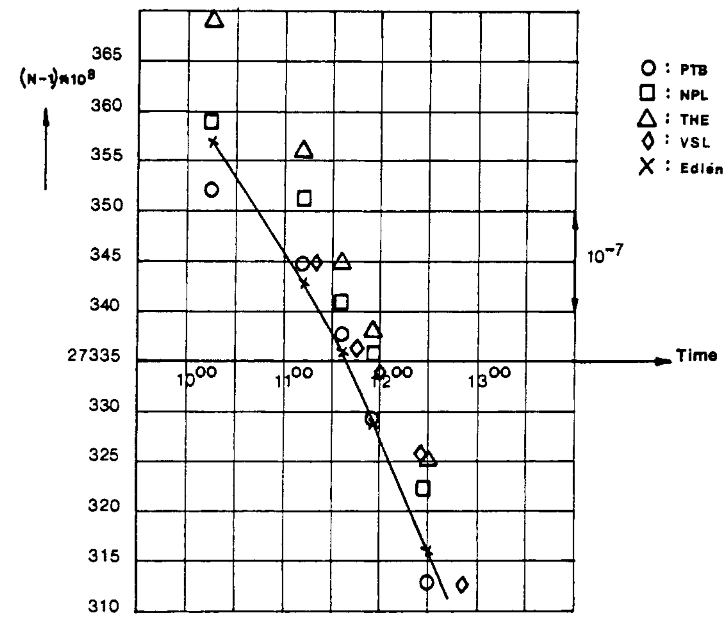


**Fig. 8.** Measurements of air refractive index on 10 June, 1983

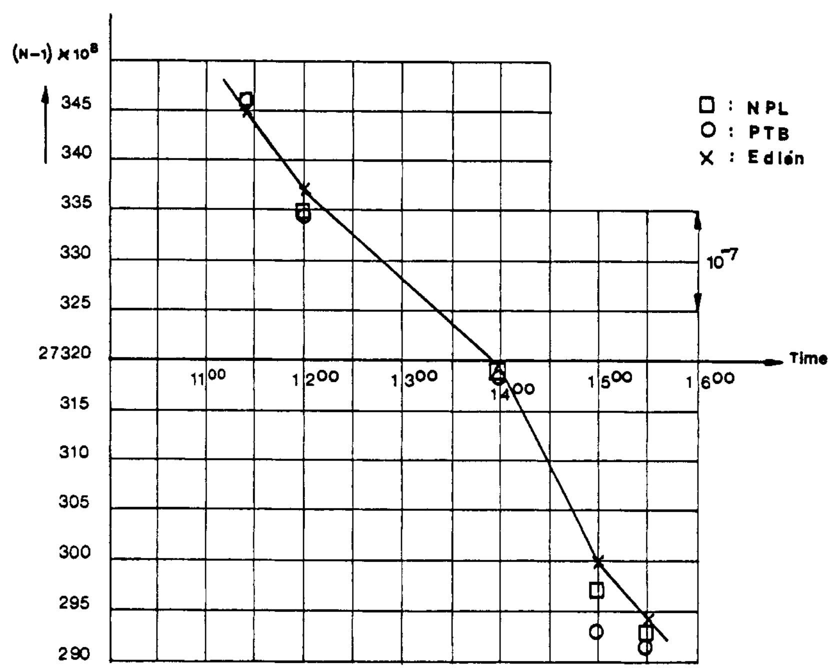


**Fig. 9.** Direct comparison NPL-PTB

a change in the humidity content of the air sample as mentioned earlier.

### *4.3. Changes of $CO_2$ Content During the Measuring Period*

The $CO_2$ content in the air of the laboratory is shown plotted in Fig. 11. There is a considerable increase of $CO_2$ content over the day; this can be related to the number of people (7–8) taking part in the experiments in a 300 m$^3$ volume room which was air-controlled by a closed circuit. The influence of the $CO_2$ content increase can be calculated from the formula given in Sect. 4.1. This gives an increase in N of 1.4 parts in $10^8$ for a 100 ppm increase in $CO_2$ content.

### *4.4. Uncertainty of the Results*

The uncertainty in the calculated values of N arises from two main sources: the realiability of the equation and the accuracy of the sensors used to

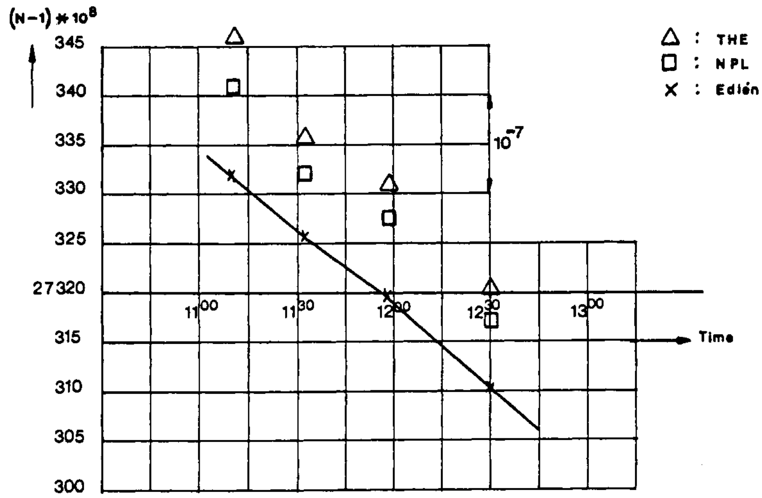


Fig. 10. Direct comparison NPL-THE

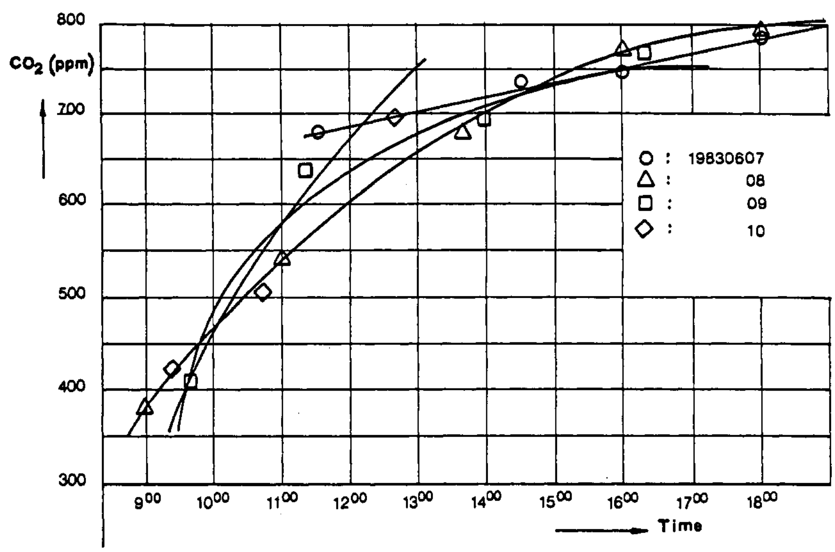


Fig. 11. $CO_2$ content in cabinet during experiments

measure air temperature, barometric pressure, humidity and $CO_2$ content. The total uncertainty associated with the two sources is calculated to be $\pm 8$ parts in $10^8$. The uncertainty in the measured values of the refractive index for each of the four instruments is given below:

NPL: $\pm$ 2.2 parts in $10^8$
PTB: $\pm$ 1.0 parts in $10^8$
THE: $\pm$ 4.0 parts in $10^8$
VSL: $\pm$ 6.0 parts in $10^8$.

These are instrumental uncertainties. Further uncertainties will occur when differential temperature measurement is used. The magnitude of these errors will be at least 3 parts in $10^8$ and will depend upon the measurement situation. All uncertainties given correspond to a 95% confidence interval ($2\sigma$).

## 5. Conclusions

From these results it may be concluded that most measured values cover the aim of the project. The measurements in some cases indicated imperfections of the set-up which can be easily modified, e.g. the air-inlet system of the THE refractometer has been evaluated since that time [5].

Direct comparisons of refractometers show an agreement better then 5 parts in $10^8$ and thus significantly better than the accuracy of 1 part in $10^7$ aimed at. The comparison of measured and calculated values proves that the model accuracy of Edlén's formula is better than 5 parts in $10^8$, provided that correction is carried out for $CO_2$ content. If, however, the carbon dioxide content is not taken into account, as with commercially available laser interferometers, errors, which could exceed 1 part in $10^7$, are possible.

It must be stressed that during the experiments the atmosphere was not contaminated to any extent, and in industrial environments a much larger disturbance might be expected.

The final conclusion may be that the intercomparison has provided the information required for the development of instruments for use in industrial applications where a length-measurement precision of 1 part in $10^7$ is required.

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## References

1. B. Edlén: Metrologia **2**, 71–80 (1965)
2. P. Schellekens: BCR-Report on the measurements of the refractive index of air using interference refractometers. Community Bureau of Reference, Commission of the European Communities, Brussels, 1984 (in press)
3. M. Downs, K. Birch: Precis. Eng. **5**, 105–110 (1983)
4. G. Wilkening, PTB: to be published
5. Ch. Verrostte: *Some Experiments with a New Air-Refractometer.* University Report WPB-0051, 1984, Eindhoven