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# Design and Results of a New Interference Refractometer Based on a Commercially Available Laserinterferometer

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A new type of interference refractometers, based on a commercially available laserinterferometer, has been developed. The design and behaviour of this refractometer are presented in this paper. A review of measurements on stability, absolute measuring accuracy and a comparison with calculated values of the refractive index of air will be given. The maximum uncertainty has been evaluated as 5 parts in  $10^8$ . This refractometer can be very useful for the accurate determination of the refractive index of air and other gases and also for the calibration of automatic compensators of commercially available laserinterferometers.

## 1. INTRODUCTION.

Laserinterferometry has been used for many years as a useful tool for the accurate measurement of length, angle and straightness. Today laserinterferometers are the basic instruments for the calibration of coordinate measuring machines and other accurate length measuring instruments.

There is also a growing need for the very accurate determination of errors in scales, straightness and squareness of coordinate measuring machines, because these data are now used for software error correction in these instruments.

Laserinterferometers are also used as measuring systems in very accurate production- and measuring machines. When using interferometry the measured length  $L$  is generally calculated from

$$L = \frac{k \lambda_v}{Np} \quad (1)$$

where -  $k$  is the number of pulses counted,  
-  $\lambda_v$  the vacuum wavelength of the lasersource,  
-  $N$  the refractive index of air and,  
-  $p$  a numerical factor usually two or four, depending on the optical and electronic properties of the instrument.

The relative uncertainty of  $L$  can be calculated from Eq. (1):

$$\frac{\Delta L}{L} = \frac{\Delta k}{k} + \frac{\Delta \lambda_v}{\lambda_v} - \frac{\Delta N}{N} \quad (2)$$

Earlier in these Annals we have published information about the determination of the accuracy of  $k$  and  $\lambda_v$  [1,2]. It may be concluded from that information and Eq. (2) that the relative accuracy of  $L$  is limited by the accuracy  $\frac{\Delta N}{N}$ . Today for the most accurate measurements  $\frac{\Delta L}{L} < 10^{-7}$  is demanded which means that at least the same accuracy for  $N$  is required. For most commercially available laserinterferometers  $N$  has to be calculated from the measured values of pressure, temperature and humidity using Edlén's equation [3]. Somewhat modified in the air this equation can be written as:

$$N-1 = \frac{D \cdot 0.104127 \times 10^{-4} \cdot P}{1 + 0.3671 \cdot 10^{-2} \cdot T} - 0.42063 \cdot 10^{-9} \cdot F \quad (3)$$

where -  $D = 0.27651756 \cdot 10^{-3} (1 + 0.54 \cdot 10^{-6} (C-300))$ ,  
-  $P$  the pressure of the air in Pa,  
-  $T$  the airtemperature in  $^{\circ}C$ ,  
-  $F$  the watervapour pressure in Pa and,  
-  $C$  the  $CO_2$  content in ppm.

Commercially available laserinterferometers with electronic correction for changes in  $N$ , the so called automatic compensation, use a constant value for  $C$ , normally 300 ppm. From Eq. (3) the influence of errors can be calculated. For sake of completeness they are given in Eq. (4):

$$\begin{aligned} \frac{\Delta N}{\Delta P} &= 2.67 \cdot 10^{-9} \text{ Pa}^{-1} \\ \frac{\Delta N}{\Delta T} &= -9.20 \cdot 10^{-7} \text{ K}^{-1} \\ \frac{\Delta N}{\Delta F} &= -4.21 \cdot 10^{-10} \text{ Pa}^{-1} \\ \frac{\Delta N}{\Delta C} &= 1.45 \cdot 10^{-10} \text{ ppm}^{-1} \end{aligned} \quad (4)$$

Since the model-accuracy of Edlén's equation has been claimed to be  $5 \cdot 10^{-8}$  [3], it will be very difficult to reach the  $10^{-7}$  level for  $\frac{\Delta N}{N}$ , due to errors in the determination of  $P$ ,  $T$ ,  $F$  and  $C$ . To get a more accurate measurement of  $N$  interference refractometers have been built in recent years [4,5,6]. By these

refractometers the refractive index including the contribution of all constituents in the air is measured. As can be expected this method gives better results than the Edlén-method. This has been confirmed by an international project on the comparison of interference refractometers. In this project, supported by BCR-EEC, Brussels, four refractometers developed in different countries are compared. The measured values of  $N$  are also compared with calculated values using Edlén's equation. The complete results are published now [6].

The Dutch participants in this project have decided to redesign their refractometers and to build improved instruments based on the same concept: using commercially available laserinterferometers. Two refractometers have been built based on the same principle but using different lasersources and counting systems [7] of a well known manufacturer. It should be noted that these refractometers can be used also with other lightsources and counting systems.

We have compared these refractometers and, separate, calculated  $N$  from Eq. (3). In this way we could also determine the differences between measured and calculated values of  $N$ . Finally, the results of the calibration of automatic compensators of laserinterferometers performed with this refractometer are given.

## 2. DESIGN OF THE INTERFERENCE REFRACTOMETER.

The refractometer has been based on a symmetric double-beam interferometer. One of the beams is passing through a sample-chamber which can be filled with the medium to be measured, normally air. The other beam is passing a vacuum chamber as a reference.

Vacuum and air chambers are drilled in an aluminium block, sizes  $70 \times 70 \times 400$  mm. The chambers are closed by optical windows. Connections for filling and pumping out the chambers are provided on the aluminium block. Two sensors for temperature measurement are mounted in the sample-chamber. The optics for beam-splitting, beam-bending and reflection are mounted separately on a common base together with the aluminium block and the lasersource. These optics are not standard components of the laserinterferometer used, but are available commercially.

The principle of the refractometer is shown schematically in Fig. 1. The quarter-wave plate is only necessary if a polarizing beam-splitting prism is used as is the case in our refractometers.

It should be noted that the prisms for beamsplitting, beambending and also the lasersource can be fine adjusted to optimise the interference signal for the counting system.

The method of measurement is very simple: first the chambers are evacuated to a sufficient low pressure, after which the medium - air - is admitted slowly into the sample-chamber through a filling station. The changing optical path has been measured by the interferometer during the filling period, normally a few minutes.

It can easily be proved that in the case of constant optical paths outside the aluminium block the refractive index can be calculated from:

$$N - 1 = \frac{k \cdot \lambda_v}{p \cdot L_T} \quad (5)$$

where -  $k$  is the number of counts due to the changing optical path in the sample-chamber,  
-  $p$  a numerical factor, and  
-  $L_T$  the actual length of the sample-chamber.

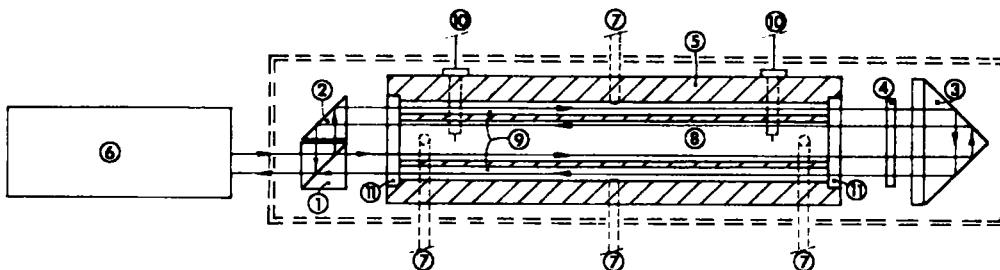


FIG. 1: PRINCIPLES OF THE REFRACTOMETER.

- 1: beamsplitting prism; 2: beambending prism;
- 3: corner-cube; 4:  $\lambda/4$ -plate; 5: aluminium block;
- 6: lasersource + counter; 7: filling and pumping connections;
- 8: sample-chamber; 9: vacuum-chamber;
- 10: temperature sensor; 11: optical windows.

By electronic extension provided with laser-source type A [7] a value  $p = 400$  has been reached while for type B [7] a value of  $p = 600$  has been used, using the build-in computer in connection with the straightness-measurement mode.

To ascertain that the air in the sample-chamber has the same humidity content as the air in an isolated volume, next called the sample-volume, the air has been circulated smoothly ( $1 \text{ l min}^{-1}$ ) from this volume through the sample-chamber. In the sample-volume we have measured also pressure, temperature and humidity. Daily we took several air samples for determination of the  $\text{CO}_2$  content and other constituents by gaschromatographic techniques. For the pressure measurement a Fortin-type mercury barometer and a metal barometer have been used. Temperature measurement in the sample-chamber, on the aluminium block and in the sample-volume are carried out by resistance thermometers. The thermometers, the Pt-100 type, are coupled to a measuring set-up according to Dauphinee [8]. In this set-up the resistance of the thermometers has been compared with a standard using a Diessehorst-compensator with null-detection. Most of the humidity measurements were carried out by measuring the dewpoint temperature  $T_D$  and from these results the water vapour pressure was calculated from Eq. (6):

$$F = 611 \cdot \exp(72.5 \cdot 10^{-3} \cdot T_D - 288.1 \cdot 10^{-6} \cdot T_D^2 + 0.79 \cdot 10^{-6} \cdot T_D^3) \quad (6)$$

where -  $T_D$  is the dewpoint temperature in  $^{\circ}\text{C}$ , and  
 -  $F$  the water vapour pressure in  $\text{pa}$ .

A wet and dry bulb psychrometer has been used also for comparison.

The actual length  $L_T$  of the sample-chamber can be calculated from  $L_T = L_0(1 + \alpha(T - T_0))$ . Here,  $\alpha$  is the linear coefficient of expansion of aluminium,  $L_0$  the length of the chamber at  $T_0$  and  $T$  the temperature of the aluminium block. The resolution of the type A refractometer has been calculated to be 4 parts in  $10^9$  and for type B 2 parts in  $10^9$ . The refractometers are connected to filling and pumping stations coupled by valves. Connections were made by thick-walled flexibel PVC tubes. Vacuum measurement was carried out by a Pirani-gauge.

The refractometer and optics are situated in a Polystyrene-cabinet in order to avoid temperature gradients in the refractometers. Fig. 2 shows a diagram of the complete refractometer set-up for the comparison of the measured and calculated values of  $N$ .

### 3. MEASUREMENTS AND RESULTS.

#### 3.1. Stability of the refractometers.

Before the refractive index measurements have been started the stability of the refractometers was studied. For this measurement both chambers have been evacuated and after equilibrium the change in optical path has been measured by the interferometer over periods of several hours. Both refractometers have shown nearly the same behaviour: a drift in  $N$  of a few parts in  $10^8$ . Fig. 3 shows a sample graph of the drift.

- 1: sample-volume; 2: temperature-measurement;
- 3: refractometer; 4: resistance thermometers;
- 5: valves; 6: filling station; 7: barometer;
- 8: dewpoint measurement; 9: Pirani-gauge;
- 10: circulationpump; 11: cabinet; 12: lasersource;
- 13: vacuum pump.

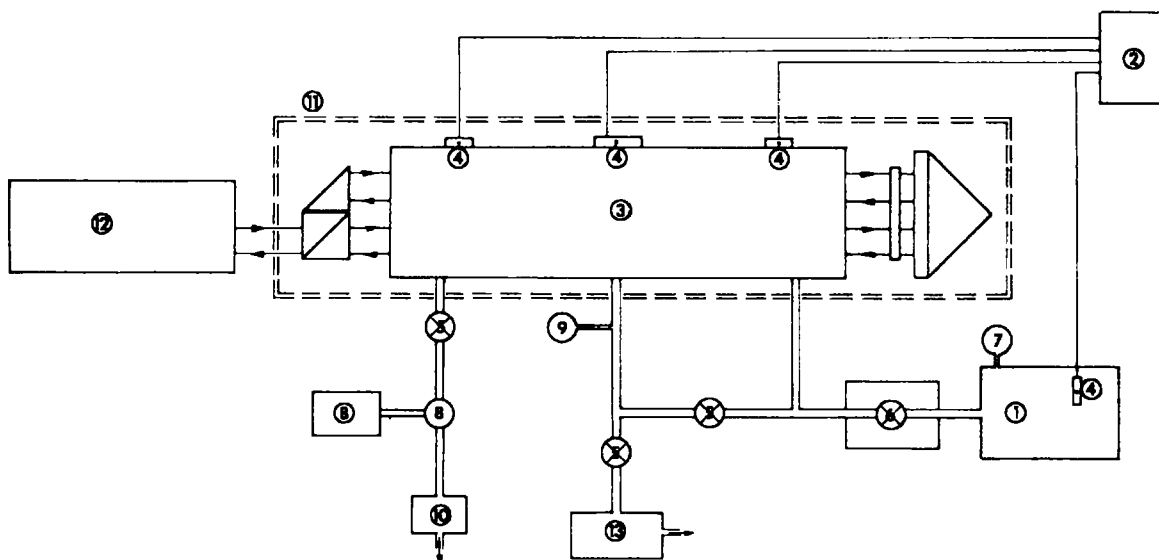


FIG. 2: MEASUREMENT SET-UP OF REFRACTOMETER FOR THE COMPARISON "MEASURED/CALCULATED" N-VALUES.

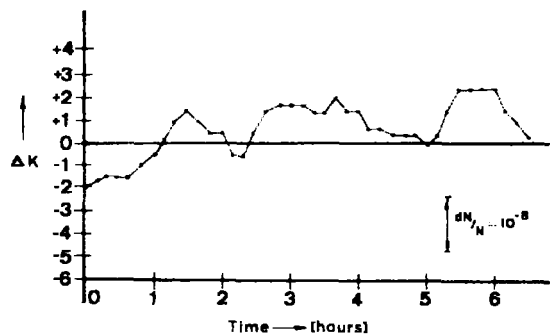


FIG. 3: STABILITY-MEASUREMENT OF THE REFRACTOMETER.

After evacuation a single measurement of  $N$  takes about 5 minutes so in that case the drift is neglectable. Continuous measurement of  $N$ , without intermediate evacuation of the sample cell, over longer periods, needs small correction for the most accurate measurements.

### 3.2. Comparison of values measured by the refractometer, with calculated values of $N$ .

In this set-up both refractometers were connected to the sample-volume. From this volume air was admitted to both sample-chambers by separate filling stations. During the measurements the air was circulated through both sample-chambers. Temperature was measured in the sample-chambers and in the sample-volume to correct the measured values of  $N$  for temperature difference between these places.

In the sample-volume also pressure, humidity and  $\text{CO}_2$ -content have been measured. From these results values of  $N$  have been calculated. Fig. 4 shows graphically some results for both refractometers. Several measurements have shown a slight tendency for the measurements of  $N$  to be higher than the calculated values as we have found also in the international comparison mentioned earlier. To confirm this, it will be necessary to improve our measurements of  $P$ ,  $T$  and  $F$ .

### 3.3. Direct comparison of the refractometers.

For these measurements both refractometers have been connected to one filling and pumping system. In this way the vacuum and air conditions were the same for both instruments. After evacuation air from the sample-volume has been admitted into both sample-chambers. Also a slight circulation of air through both chambers was accomplished. Air temperature in both sample-chambers has been measured to correct for temperature differences. Fig. 5 shows the complete set-up for the comparison of the refractometers. In this case the insulated cabinets around the refractometers are removed.

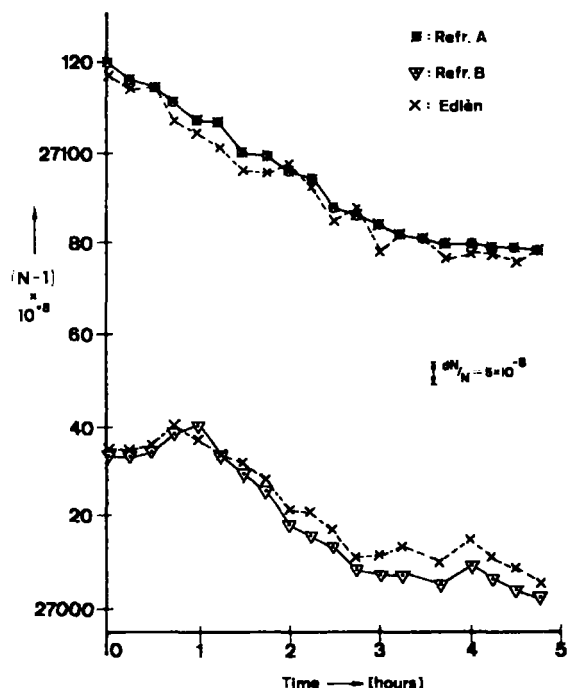


FIG. 4: MEASURED VALUES OF REFRACTIVE INDEX AND CALCULATED "EDLEN"-VALUES, RESULTS OF TWO DAYS.

In Fig. 6. the relative difference of the measured values of both refractometers is plotted.

The results shows a small difference after a longer measuring period due to the drift effect mentioned earlier. Also there can be some effect of the temperature measurement because small corrections for temperature difference have been carried out. Short time measurements have shown better results [9].

### 3.4. Role of $\text{CO}_2$ -content and other constituents.

For the most accurate measurements with laserinterferometers one has to measure the  $\text{CO}_2$ -content of the air and generally to correct for it [6]. To get a good idea about the situation in our laboratory the air has been analysed over longer periods by gaschromatography. Every day showed the same tendency: a starting value around 350 ppm and thereafter an increasing  $\text{CO}_2$ -content reaching a level between 700 and 950 ppm at the end of the day depending on the number of people in the  $400 \text{ m}^3$  volume of the

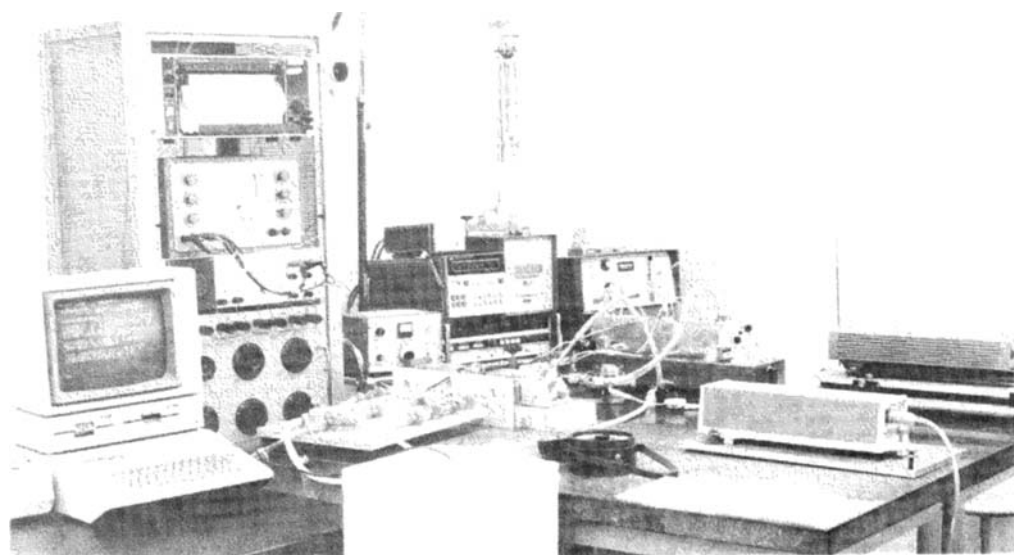


FIG. 5: SET-UP FOR DIRECT COMPARISON OF REFRACTOMETERS.

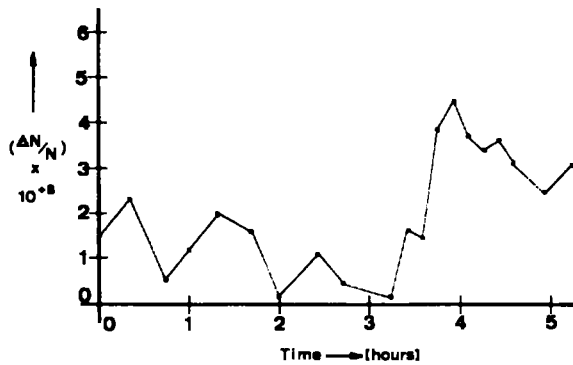


FIG. 6: COMPARISON OF REFRACTOMETERS.

laboratory. Fig. 7 shows graphically the results of the CO<sub>2</sub>-measurements over several days. Each measured point represents the average value of four analyses of the same air sample. The uncertainty of this average value has been calculated to be 15 ppm.

Different values on the same time in different days are due to the different numbers of people in the laboratory during the measuring period.

The maximum increase of around 700 ppm corresponds to  $\frac{\Delta N}{N} = 10^{-7}$ , so it is important to correct for the CO<sub>2</sub>-content. The determination of other constituents showed minor concentrations of hydrocarbons which can be neglected. During the calibration of a coordinate measuring machine at a car manufacturing plant an air sample has been analysed. The following concentrations have been detected:

Carbondioxide	: 1200 ppm
Methane	: 8 ppm
Ethane	: 1 ppm
N-Butane	: 2 ppm
Octane	: 2 ppm
Propane	: 1 ppm
N-Pentane	: 0-1 ppm

Again a high concentration of CO<sub>2</sub> has been detected but very low the concentrations of other gases. Much higher concentrations have been reported in comparable environmental conditions [5]. The following concentrations correspond to a relative change of  $10^{-7}$  in N each:

Carbondioxide	: 700 ppm
Methane	: 595 ppm
Ethane	: 205 ppm
Propane	: 130 ppm
N-Butane	: 98 ppm
Octane	: 50 ppm
N-Pentane	: 72 ppm

It may be concluded that in the car manufacturing plant situation only the carbondioxide concentration has a significant influence on the measuring accuracy.

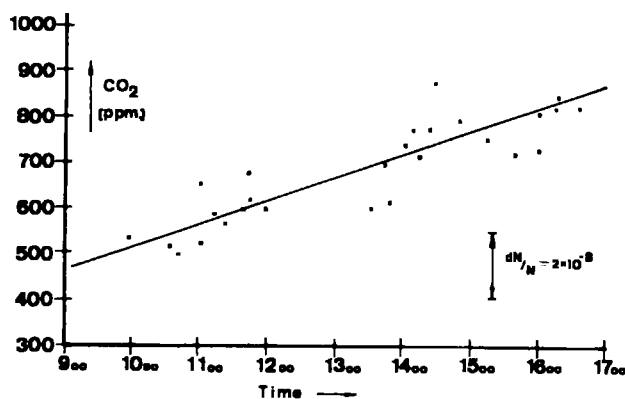


FIG. 7: DAILY BEHAVIOR OF CO<sub>2</sub>-CONTENT.

#### 4. ADDITIONAL APPLICATIONS.

The interference refractometers described can be used also for the determination of the refractive index of other gasses at

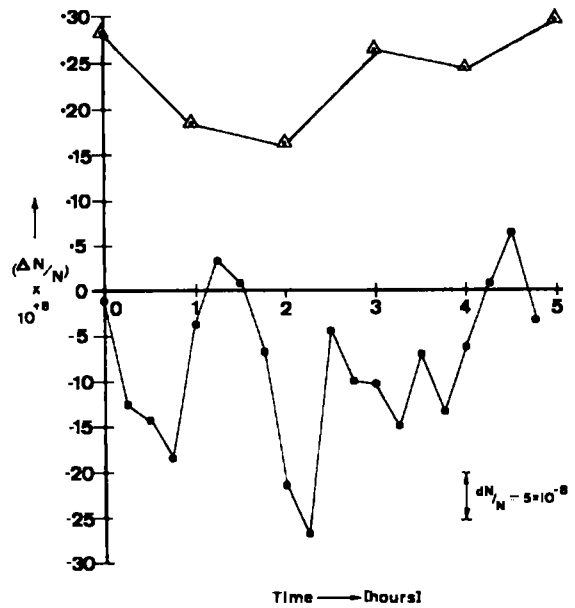


FIG. 8: COMPARISON OF MEASURED VALUES OF AUTOMATIC COMPENSATOR AND REFRACTOMETER.

$\lambda=633\text{nm}$ . In this case an extra pressure sensor has to be connected to the sample-chambers so pressure and temperature conditions can be measured.

A very important application will be the use of the refractometer as a calibration set-up for automatic compensators often used in commercially available laserinterferometers. Some compensators were verified against our refractometers. In this set-up the sensors of the compensator have been positioned in the sample-volume. The refractive index of air from this volume has been measured separately by the refractometer and the compensator. After correction for the temperature-difference between sample-volume and sample-chamber the measured values of N have been compared. Fig. 8 presents the result of two of these measurements as the relative difference between refractometer and compensator values. The compensators verified are made by the same manufacturer [7].

The relative differences are below 3 parts in  $10^7$  mainly due to measurement errors of the sensors of the automatic compensators. The manufacturer claims a maximum uncertainty of 5 parts in  $10^7$ .

#### 5. DISCUSSION.

The interference refractometers described here have shown a stability better than 5 parts in  $10^8$  over several hours. At least a part of this drift can be explained from small temperature-effect on the mechanical structure for the adjustment of the optics. Direct comparison showed also relative differences less than 5 parts in  $10^8$ . Part of these differences are due to small errors in the temperature-measurement in the sample-chambers. The uncertainty in this measurement was less than 0.02 K which causes maximum relative errors of  $\frac{\Delta N}{N} = 2 \cdot 10^{-8}$ . Also small effect have been measured from local expansion of the optical windows arising from the vacuum-air change in the sample-chamber. These effects are evaluated now.

Comparison between measured and calculated values of N, as was shown in Fig. 4, indicating relative differences up to 8 part in  $10^8$ . Part of these differences can be explained from the uncertainty in the calculated value of N. The uncertainty of the pressure measurement has been determined to be at least 15 Pa and the uncertainty in the dewpoint measurement will be 0.3 K. The carbondioxide content has been calculated from interpolation between measured values. The maximum uncertainty will be less than 50 ppm. From these values and the model uncertainty of  $\pm 5$  parts in  $10^8$  the total uncertainty in the calculated values of N will be  $\pm 7$  parts in  $10^8$ . The results showed in Fig. 4 are within this range.

#### 6. CONCLUSIONS.

It may be stated that the interference refractometers developed are very useful instruments for the accurate determination of the refractive index of air and for the calibration of automatic compensators in laserinterferometers.

Relative uncertainty will be less than 5 parts in  $10^8$  even when

the instruments are used continuously for periods of several hours. The instruments can be build relatively easy because the are based on commercially available laserinterferometers. For some types it is even possible to use the refractometer parallel to the interferometer using the same lasersource [9].

It has been proved that the CO<sub>2</sub>-content plays an important role in accurate interferometric measurements and generally it is not allowed to use the standard value of 300 ppm for calculating N values. Especially in industrial environments the CO<sub>2</sub>-content can be very high but also the butane and octane content can be important [5].

We have measured a relation between the number of people and the CO<sub>2</sub>-content in our laboratory. The air in this laboratory is conditioned by closed circuit. Generally it can be stated that for interferometric length measurements with relative uncertainty less than 1 part in 10<sup>7</sup> the refractive index has to be measured by an air-refractometer.

#### 7. ACKNOWLEDGEMENTS.

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Refractometer B: laser 5518A + 5508A display.  
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