# TECS — A New Referencing Method for Spectrophotometers

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**References cited:**

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2. "MD 5 Miniature Dual-Beam Spectrograph. Dual Beams on a Single Array," brochure, American Holographic, Inc., Fitchburg, MA
3. I.J. Fritz et al., "Broad-Band Light-Emitting Diode for 1.4-2.0 µm Using Variable-Composition InGaAs Quantum Wells," IEEE Photonics Technology Letters 7, 1270-72, November 1995

## Abstract

A new referencing method for improving the baseline stability of spectrophotometers has been developed. The idea is to use two sources and two detectors, thereby establishing four optical beam paths through the system, and have each and every optical element in the system be used by at least two, overlapping and mutually referencing, beams. The two sources are modulated and the two detector signals are demodulated so that the signal components due to the four optical beams can be separated by standard signal processing means. Spectrophotometric measurements like, e.g., transmission spectra, are computed as a ratio of two ratios. The method has been dubbed *"Two of Each, Close in Space" (TECS)* and is able to provide a complete, fully spectrally resolved, reference without mechanically moving parts.

## Field of the Invention

The invention relates to apparatus and methods for referencing spectrophotometers.

## Background of the Invention

Most of the spectroscopic analyses performed in the UV, VIS, NIR and IR wavelength ranges require ratiometric measurements, i.e., the optical signal received from the sample is compared to the optical signal received from a reference standard. Ratiometric measurements, e.g., transmission, absorbance, or reflectance spectra, are important for correct qualitative and quantitative interpretation.

The main purpose of a spectrophotometric measurement is to isolate the spectral signature of the sample from the spectral characteristic of the instrument (lamp, filters etc.). This is achieved by measuring the response of the instrument to a reference standard, the material and spectral response of which are generally chosen to be as flat as possible throughout the wavelength range of interest. The spectral response measured from the sample is then ratioed by the response measured from the reference and, ideally, all spectral signatures unrelated to the sample will be canceled.

The typical instrument today is a so-called single-beam spectrometer in which the reference and the sample are measured sequentially. Typical research instruments like, e.g., many Fourier-Transform IR spectrometers, require the operator to manually put the reference into position. Instruments used for on-line process control and instruments dedicated to routine analyses typically also measure the reference sequentially but may have electro-mechanical means for moving the reference into position.

Practical spectrometers are not perfectly stable but show response drifts due to, e.g., room temperature variations. The instability of an instrument can be observed by ratioing the response signals from two consecutively measured references; typically, instruments will deviate from the ideal "100% transmission baseline" anywhere in the range from 0.1% to 10%. In practice, the stricter the requirements of the application, the more frequently the reference measurement needs to be repeated. On typical FT-IR spectrometers, for example, qualitative analyses may require only a single reference per day whereas some quantitative analyses may require a new reference every other minute. Because of practical limitations as to how fast the reference can be moved, baseline instability turns out to be a limiting factor for accuracy in single-beam instruments.

In the early days of optical spectroscopy researchers tried to build double-beam "spectrophotometers" in which the sample and the reference could be measured quasi-simultaneously. Typically, two separate optics, which were mirror images of each other, would pick up radiation emitted from a single source, direct the two beams to the sample and the reference, then pick up the two beams and direct them to a rotating mirror which would transfer the beams to a single detector (e.g. [1]). In practicee, however, double-beam instruments turned out to be inferior to single-beam spectrometers with respect to baseline stability and reliability and the idea has been largely abandoned to this date. The reason for the failure of the early double-beam instruments was that the two separate optical paths were just that, separate. If one optical element got misaligned, or fingerprinted, and its respective mirror image element did not undergo the exact same change in its spectral response characteristic then the accuracy of the baseline of the instrument was compromised.

## Summary of the Invention

This invention provides a method for achieving a complete, quasi-simultaneous, long-term stable, fully spectrally resolved reference in a spectrophotometer without mechanically moving parts. As a result of using this invention, the baseline stability of spectrophotometric measurements can be improved by several orders of magnitude compared to current single-beam instruments and highly accurate measurements can be performed reliably, even in uncontrolled or hostile environments. Important applications of this invention include IR process control sensors and IR biomedical home monitoring sensors.

In the following, the notation of Tab.I will be used for the four optical beams and their respective electrical counterparts. Also, a transmission measurement will be used as an example. The invention, however, can be applied to other spectrophotometric measurements like absorbance or reflectance.

| Tab.I | Detector I<br>Instrument | Detector II<br>Sample, Reference |
|---|---|---|
| Source #A<br>Sample | $S^{I}_{A}$ | $S^{II}_{A}$ |
| Source #B<br>Reference | $S^{I}_{B}$ | $S^{II}_{B}$ |

The basic idea behind this invention is to use two sources and two detectors, thereby defining four optical beam paths through the system, and have each and every optical element in the system be used by at least two, overlapping and mutually referencing, beams. The transmission spectrum is computed as a ratio of two ratios:

$$T = \frac{\left(\frac{S_A^{II}}{S_A^{I}}\right)}{\left(\frac{S_B^{II}}{S_B^{I}}\right)} \qquad (1)$$

Two beams are mutually referencing whenever they form a ratio in Eq.1, e.g., beam $S^{II}_{A}$ references with $S^{I}_{A}$ and $S^{II}_{B}$ but not with $S^{I}_{B}$. In the following, $S^{II}_{A}$ will be called the sample beam; $S^{II}_{B}$ will be called the reference beam; and $S^{I}_{A}$ and $S^{I}_{B}$ will be called the instrument beams. The ratio $S^{II}_{A}/S^{II}_{B}$ in Eq.1 is the spectrophotometric ratio and provides spectral information about the sample. The instrument beams do not provide spectral information about the sample but do provide spectral information about the baseline of the instrument. As will be explained in detail later, referencing can take place between all possible pairs of referencing beams in actual TECS spectrophotometers.

The closer the two beams of a referencing pair are to each other in space, i.e, the more they overlap at the optical surfaces and in the free space between optical surfaces, the less sensitive the baseline of the instrument will be with respect to dust, absorbers in the atmosphere, mechanical misalignments etc. Practically speaking, TECS spectrophotometers should be designed for maximum beam overlap throughout the system in order to increase baseline stability and reliability and in order to avoid expensive baseline protection mechanisms like purging.

Of course, given the very nature of the measurement, the sample beam and the reference beam need to be separated in space somewhere in the system so that one beam can hit the sample and the other the reference. In many applications, however, the area of separation can be restricted to a small space in the immediate vicinity of the sample which, in turn, can relatively easily be protected from environmental influences. In particular, the area of separation can be placed inside an optical glass to fully benefit from the uniformity and environmental stability of the glass. The state of the art of optical design makes available to optical engineers and spectroscopists a large variety of different designs by which separation of sample and reference can be achieved in a protected environment. In practice, choice of design will depend on the physical characteristics of the sample, the type of spectrometer used (e.g. FT-IR, grating), and the type of experiment (e.g. transmission, reflection). In the following, three particularly useful designs will be described in detail.

## Brief Description of the Drawings

FIG.1 shows a preferred embodiment of TECS for a diffuse reflection experiment and an LED array based instrument.

Fig.2 shows a preferred embodiment of TECS for a transmission experiment and a detector array based instrument.

Fig.3 shows another embodiment of TECS for a transmission experiment and a detector array based instrument.

## Detailed Description of the Invention

A preferred embodiment of TECS for a diffuse reflection experiment using an LED array based instrument is shown in Fig.1.

Two LED arrays (1A and 1B) that may or may not be packaged into a single package, emit light onto a grating (2) and monochromatic images (4A and 4B) are formed in the exit slit (5) of monochromator (3). The two arrays (1A) and (1B) have identical number of emitters and identical spectral coverage. Mirror (6) and lens (7) direct the two beams onto a beamsplitter (8) where a small portion of the radiation power is reflected and is directed through lens (9) to detector I (10). Detector I (10) detects the two instrument beams $S^I_A$ and $S^I_B$. Lens (9) is a Kohler lens that images the aperture of beamsplitter (8) onto detector I (10), i.e., the two beams are not separated at detector I (10) but are overlapping. Most of the optical power, however, is transmitted through beamsplitter (8) and lens (11) and mirror (12) to an immersion lens (13) contacted by the sample (14). Lens (13) is dimensioned big enough to allow significant separation of the sample beam and the reference beam to occur only within the glass of (13) and is made out of, e.g., Bk-7, fused silica, or sapphire. The separation of the LED images (4A) and (4B) in the exit slit (5) is used to focus the reference beam onto the reference standard (15) which is immersed and protected in lens (13) whereas the sample beam is directed onto the sample (14). The diffusely reflected optical signals from both the sample (14) and the reference (15) are collected by pick-up optics (16) and concentrated onto detector II (17). Detector II (17) is used to detect both the sample and the reference signal whereas detector I (10) is used to detect the two instrument beams. The detector signals are amplified by pre-amps (18) and (19) and fed into a signal processing electronics (20) typically comprised of additional amplifiers, a multiplexer, sample & hold, an A/D converter, digital signal processing, a central processing unit, and associated digital hardware.

The optical signals emitted by the two arrays ($S_A$ and $S_B$) are modulated in time, e.g., in a simple on-off fashion in which only a single LED is on at a time. Electronics (20) is used to demodulate the two detector signals ($S^I$ and $S^{II}$) and separate the four components, $S^I_A$, $S^I_B$, $S^{II}_A$, $S^{II}_B$. Phase-sensitive demodulation is preferred and is achieved by having control electronics (21) provide timing signals (21a) to both the LED driver (22) and the signal processing electronics (20). The TECS double ratio (Eq.1) is then computed by the CPU in (20) and the resulting diffuse reflection spectrum is displayed on display (23).

At any one particular wavelength, the system shown in Fig.1 will produce the following TECS double ratio result:

$$Reflectance = \frac{\left(\dfrac{S_A^{II}}{S_A^{I}}\right)}{\left(\dfrac{S_B^{II}}{S_B^{I}}\right)} = \frac{\dfrac{E_{\#A}\cdot T_{\#A\text{-}BS}\cdot T_{BS,A}\cdot T_{BS\text{-}Sample}\cdot R_{Sample}\cdot T_{Sample\text{-}\#2}\cdot R_{\#2}}{E_{\#A}\cdot T_{\#A\text{-}BS}\cdot R_{BS,A}\cdot T_{BS\text{-}\#1,A}\cdot R_{\#1}}}{\dfrac{E_{\#B}\cdot T_{\#B\text{-}BS}\cdot T_{BS,B}\cdot T_{BS\text{-}Ref}\cdot R_{Ref}\cdot T_{Ref\text{-}\#2}\cdot R_{\#2}}{E_{\#B}\cdot T_{\#B\text{-}BS}\cdot R_{BS,B}\cdot T_{BS\text{-}\#1,B}\cdot R_{\#1}}} \approx \frac{R_{Sample}}{R_{Ref}} \quad (2)$$

*assuming that:*

$$T_{BS,A} \approx T_{BS,B} \quad (2.a) \qquad T_{BS\text{-}Sample} \approx T_{BS\text{-}Ref} \quad (2.c)$$

$$R_{BS,A} \approx R_{BS,B} \quad (2.b) \qquad T_{Sample\text{-}\#2} \approx T_{Ref\text{-}\#2} \quad (2.d)$$

$$T_{BS\text{-}\#1,A} \approx T_{BS\text{-}\#1,B} \quad (2.e)$$

where:

| | | | |
|---|---|---|---|
| $E_{\#A}$ | LED emission, array A | $E_{\#B}$ | LED emission, array B |
| $T_{\#A\text{-}BS}$ | transmission, from array A to beamsplitter | $T_{\#B\text{-}BS}$ | transmission, from array B to beamsplitter |
| $T_{BS,A}$ | transmission through beamsplitter, array A | $T_{BS,B}$ | transmission through beamsplitter, array B |
| $T_{BS\text{-}Smple}$ | transmission, from beam-splitter to sample | $T_{BS\text{-}Ref}$ | transmission, from beam-splitter to reference |
| $R_{Sample}$ | diffuse reflection of the sample | $R_{Ref}$ | diffuse reflection of the reference |
| $T_{Smple\text{-}\#2}$ | transmission, from sample to detector II | $T_{Ref\text{-}\#2}$ | transmission, from reference to detector II |
| $R_{BS,A}$ | reflection of beam-splitter, array A | $R_{BS,B}$ | reflection of beam-splitter, array B |
| $T_{BS\text{-}\#1,A}$ | transmission, from beam-splitter to detector I, array A | $T_{BS\text{-}\#1,B}$ | transmission, from beam-splitter to detector I, array B |
| $R_{\#2}$ | respnsivity of detector II | $R_{\#1}$ | responsivity of detector I |

The "≈" sign in Eqs.2a-e is used to allude to an important point, viz. the difference between ratiometric accuracy and stability. If accuracy is required, then the optical system needs to be designed in such a way that each pair of parameters in Eqs.2a-e are equal, e.g., $T_{BS,A}=T_{BS,B}$, at all wavelengths. If stability is required, then the parameters do not need to be equal and the optical system only needs to be designed to assure stability of the ratio of each pair of parameters in Eqs.2a-e, e.g., $T_{BS,A}/T_{BS,B}$, over time. Many spectrophotometric applications, in fact, only require stability and designers of TECS spectrophotometers can use this requirement to their favor.

All optical elements in Fig.1 are being used by two, overlapping and mutually referencing, beams. The beam separation at the exit slit (5) does not violate the TECS principle because $T_{*A-BS}$ and $T_{*B-BS}$ are referenced out between detectors I and II, and not between sources A and B (cmp. Eq.2). Generally, for systems similar to the one shown in Fig.1, beams do not need to be close in space before the beamsplitter but only at, and after, the beamsplitter.

A preferred embodiment of TECS for a transmission experiment using a detector array based instrument is shown in Fig.2.

The radiation from two broad band sources (51A) and (51B) that may or may not be packaged into a single package, is collected by a lens (52) and directed towards a beamsplitter (53) where a small portion of the radiation power is reflected and directed through lens (54) to entrance slit (55) of spectrograph (57). Lens (54) is a Kohler lens that images the aperture of beamsplitter (53) onto entrance slit (55), i.e., the two beams are not separated at entrance slit (55) but form an overlapping image (56). Polychromatic image (56) is wavelength-dispersed and imaged by concave grating (58) onto detector array I (59I). Most of the optical power, however, is transmitted by beamsplitter (53) and enters an environmental protection box (70) through a window (60). Inside

(70), the sample and the reference beams separate; the sample beam (from source 51A) is focussed by lens 61 onto the sample (62) and the reference beam (from source 51B) is focussed onto the reference standard (63). The two beams overlap again at lens (64) and leave protection box (70) through window (65). Mirror (66) and lens (67) direct the radiation towards a second entrance slit (68) of spectrograph (57). Spectrograph (57) is a so-called "dual-beam" spectrograph similar in its optical design to, e.g., the MD-5 model from American Holographic [2]. The main advantage of incorporating a "dual-beam" spectrograph into a TECS spectrophotometer is the significant reduction of optical and optomechanical parts compared to using two separate spectrographs. In addition to conventional "dual-beam" spectrographs having two entrance slits and a single detector array, spectrograph (57), however, also has two detector arrays making it a fully operational dual-beam subsystem under the TECS method. The two detector arrays (59I) and (59II) of spectrograph (57) are arranged in a standard, so-called "over & under" arrangement which is accomplished by locating the polychromatic images (56) and (69) at different optical heights in entrance slits (55) and (68), respectively. As a result, image (56) is dispersed and imaged onto detector array (59I) while image (69) is dispersed and imaged onto detector array (59II).

The driver electronics for broad band sources (51A) and (51B) and the readout and signal processing electronics for detector arrays (59I) and (59II) are standard components and are not shown in Fig.2. Broad band sources (51A) and (51B) are modulated in time and modulation preferrably is in a simple, alternating on-off fashion. Sources (51A) and (51B) are preferrably solid-state sources with fast response times, e.g., broad-band LEDs in the near IR wavelength range [3]. Detector arrays

(59I) and (59II) are preferrably; integrated monolithically; packaged into a single package; and have multiplexed readout electronics in the package. Phase-sensitive demodulation of the spectral signals detected by arrays (59I) and (59II) is preferred. After demodulation, the TECS double-ratio (Eq.1) is computed using standard signal processing means (cmp. discussion of Fig.1 above). The different optical and electro-optical transfer factors in the system transfer function of Fig.2 cancel, like they did in Eq.2 for Fig.1.

Significant advantages of the TECS method as demonstrated in Fig.1 and Fig.2 above include:

1. source emission drift referenced out simultaneously
2. detector responsivity drift referenced out in fast time-multiplex
3. fully spectrally resolved reference
4. long-term stability of optical components not required
5. completely referenced system, all optical components included
6. spectroscopic reference standard permanently sealed and protected
7. no mechanically moving parts

Another embodiment of TECS for a transmission experiment and a detector array based instrument is shown in Fig.3.

Radiation emitted from a tungsten source (101) is collected by a lens (102) and directed towards a mirror (103) and beamsplitter (104). A chopper blade (100) with two staggered slots (100A) and (100B) that are field stops in the optical paths of the system, is used to provide two spatially separated, modulated sources. A portion of the radiation power is reflected by beamsplitter (104) and directed through lens (105) and slot (100I) to mirror (106) and beamsplitter (107) where part of the optical power is reflected and focussed by lens (108) onto entrance slit (109) of spectrograph (111). Grating (110) of spectrograph (111) disperses and focusses the radiation onto detector array (112). Most of the optical power is transmitted by beamsplitter (104) and enters an environmental protection box (121) through a window (115). Inside (121), the sample and the reference beams separate; the sample beam (from slot 100A) is focussed by lens (116) onto the sample (117) and the reference beam (from slot 100B) is focussed by lens (116) onto the reference standard (118). The two beams overlap again at lens (119) and leave protection box (121) through window (120). Mirror (122) and lens (123) direct the radiation through slot (100II) and towards beamsplitter (107) where the sample and the reference beams are recombined with the instrument beams. Lenses (105) and (123) are Kohler lenses that, in combination with lens (108), image the apertures of beamsplitter (104) and mirror (122), respectively, onto entrance slit (109). The driver electronics for broad band source (101), the control electronics for chopper blade (100), and the readout and signal processing electronics for detector array (112) are standard components and are not shown in Fig.3. Detector array (112) preferrably is integrated monolithically and has multiplexed readout electronics in the package.

The main difference between the TECS spectrophotometers shown in Fig.2 and Fig.3 is that the system shown in Fig.3 uses a chopper blade (100) with slots (100A), (100B), (100I) and (100II) to apply the TECS referencing method to "conventional" systems with a single source (101), single entrance slit (109), and single detector array (112). The system shown in Fig.3 provides a relatively straightforward and cost-effective way to upgrade existing instruments with the TECS referencing method because many of today's instruments use chopper blades for source modulation anyway. In Fig.3A, slots (100A), (100B), (100I) and (100II) are arranged in such a way that the four signals $S_A^{II}$, $S_A^{I}$, $S_B^{II}$, and $S_B^{I}$, are modulated in time, i.e., one signal is detected at a time. An alternative is shown in Fig.3B where the four beams are modulated in frequency which is accomplished by having the openings of the four slots subtend different angular periods. Phase-sensitive demodulation of the spectral signals detected by detector array (112) is preferred and can be realized by using either time modulation and gated amplifiers, or frequency modulation and lock-in amplifiers. After demodulation, the TECS double-ratio (Eq.1) is computed using standard signal processing means (cmp. discussion of Fig.1 above). The different optical and electro-optical transfer factors in the system transfer function of Fig.3 cancel, like they did in Eq.2 for Fig.1.

The systems shown in Figs.1-3 are merely examples of three particularly useful designs of TECS spectrophotometers. Modifications are possible to the optical design of the relay optics, the spectrographs, and the sample/reference optics; as well as to the modulation scheme of the sources. Modifications to, e.g., the system in Fig.2 include: the dual beam spectrograph could be replaced by two separate Cerny-Turner spectrographs with one entrance slit and one detector array each, or, the two broad-band LED sources could be modulated in frequency instead of in time, or, the two broad band LEDs could be replaced by a tungsten bulb and a chopper wheel with two staggered slots if mechanical movement were tolerable, or, the two broad-band LED sources could be replaced by a tungsten bulb and a modulated optical filter transmitting only one of two states of polarization at a time in which case a polarization-sensitive beamsplitter could be used to separate and recombine the sample beam and the reference beam at the sample.

It is important to note that the TECS referencing method can be applied to all types of spectroscopic instruments and not just to instruments using diffraction gratings. Again, Figs.1-3 are merely examples of TECS spectrophotometers and the idea is simple enough that it can be applied to many alternative systems. TECS, for instance, can be applied to AOTF-based instruments or to Fourier-Transform spectrometers. In fact, TECS works for anybody who is willing to introduce a second, modulated source and a second detector into his system. By applying the TECS method, the task of designing a stable spectrophotometer is reduced to the task of designing stable sample optics.

Lastly, it is mentioned that the TECS method can have applications outside of the fields of UV, VIS, NIR or IR spectroscopy, viz. for other ratiometric measurements where the concept of "beam overlap" applies.

What is Claimed

1. A method for referencing spectrophotometers comprising the steps of:
   a) using two radiation sources and two radiation detectors, thereby establishing four optical beam paths through the system;
   b) modulating the two sources with different time functions;
   c) having each and every optical element in the system be used by at least two, overlapping and mutually referencing, beams;
   d) separating one pair of mutually referencing beams at at least one point in the system and positioning the sample into one of the two beams and the reference into the other beam;
   e) demodulating the detector signals and separating the signals due to the four optical beams;
   f) computing the spectrophotometric ratio as a ratio of two ratios.

2. The method as set forth in Claim 1, wherein the area of separation between the sample beam and the reference beam is restricted to the interior of an optical glass element.

3. The method as set forth in Claim 1, wherein the area of separation between the sample beam and the reference beam is protected by an enclosure.

4. The method as set forth in Claim 1, wherein the two radiation sources are replaced by a single source and a chopper wheel.

5. The method as set forth in Claim 1, wherein the two radiation sources are replaced by a single source and an optical filter transmitting only one of two states of polarization at a time.

6. The method as set forth in Claim 1, wherein the two radiation detectors are replaced by a single detector and a chopper wheel.

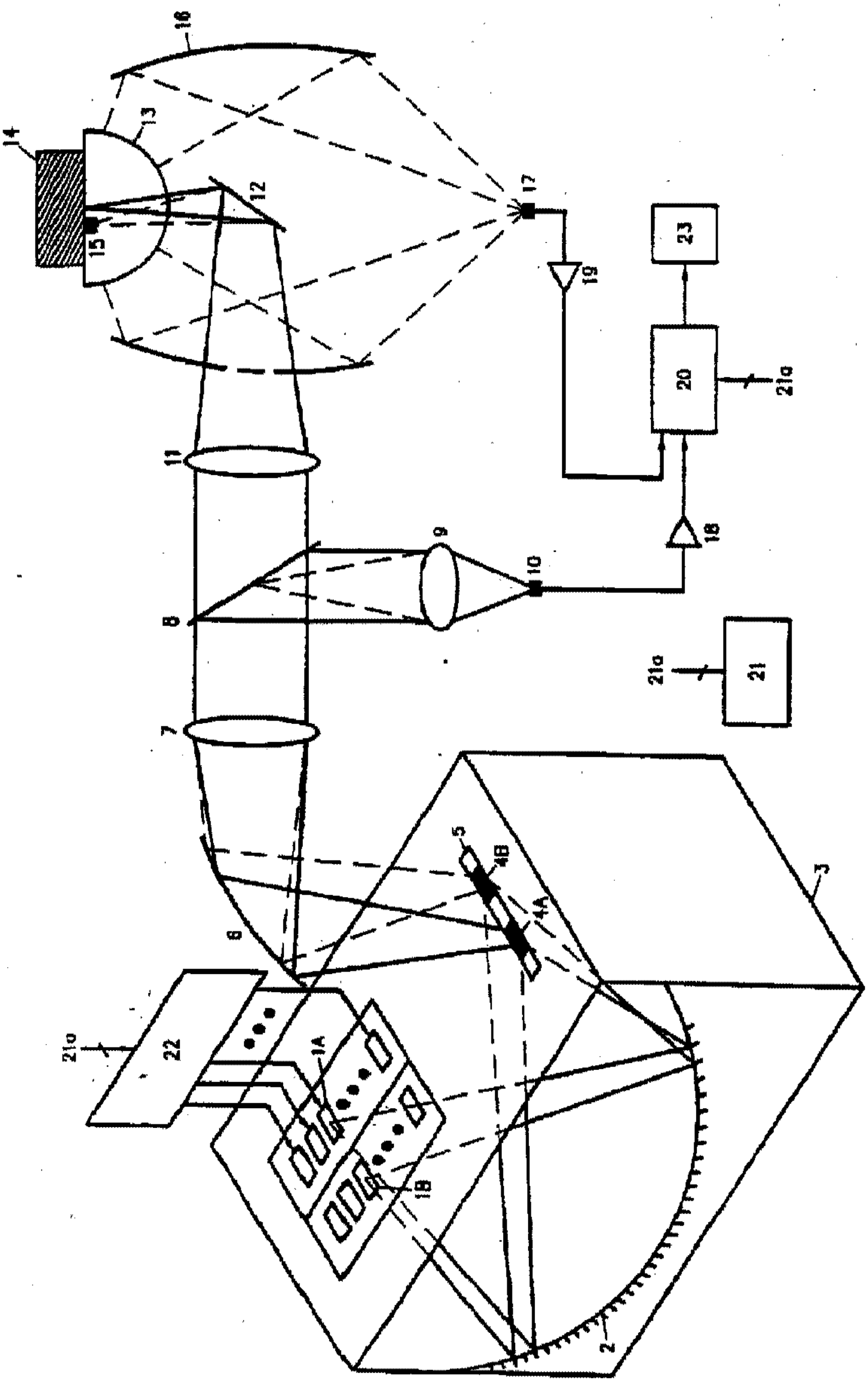
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FIG. 1

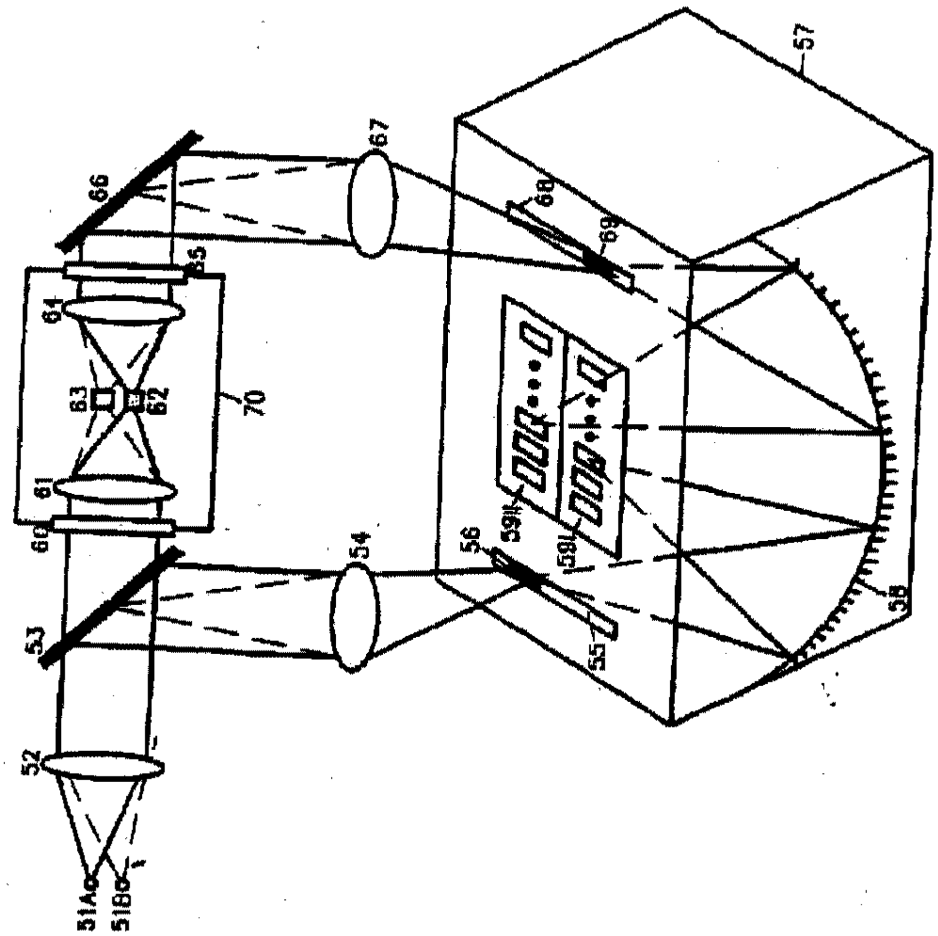


FIG. 2

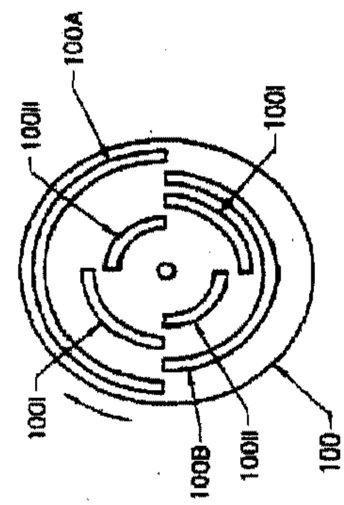


FIG. 3A

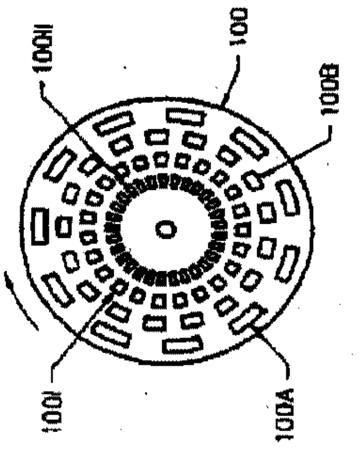


FIG. 3B

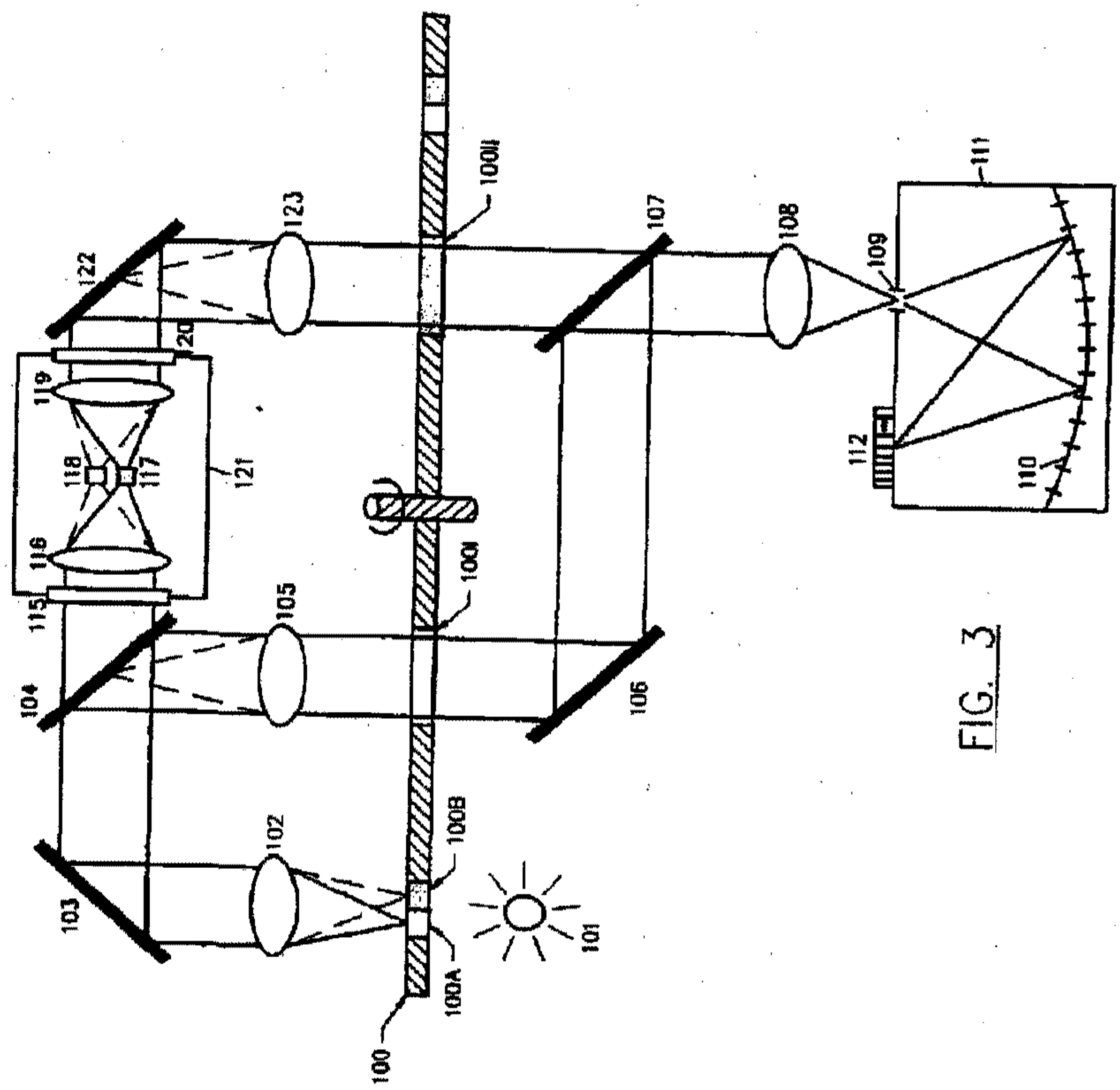


FIG. 3

WEBB FILE 2301-960681

Applicant or Patentee: Ralf Marbach
Serial or Patent No.: ______
Filed or Issued: ______
For: TECS - A New Referencing Method for Spectrophotometers

Attorney's Docket No.: 2301-960681

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described in

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TITLE OF PERSON IF OTHER THAN OWNER Vice President
ADDRESS OF PERSON SIGNING The Bourse, Building 2500, 2nd Floor
2275 Swallow Hill Road, Pittsburgh, PA 15220

SIGNATURE David E. Staudenmaier DATE 5/21/96
David E. Staudenmaier