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REFLECTANCE MEASUREMENTS

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ABSTRACT

The measurement of the reflectance of objects is essential to the programs of the Space Sciences Laboratory of the George C. Marshall Space Flight Center.

Stable thermal coatings are required to control the internal temperature of almost all objects placed in space for scientific, commercial, industrial, and military purposes. Likewise, most space objects have optical surfaces subject to degradation and contamination upon exposure to the space environment. Finally, even space objects not having these requirements cannot be allowed to contaminate those in close proximity which do. Thus reflectance measurements of coatings and optics are required to control the environment and performance on every space mission.

Spectroreflectometers are currently used to perform these measurements. These instruments are massive, well-built pieces of laboratory equipment, not optimally designed for high-volume measurement work now beginning to be required. Furthermore the equipment design is approaching twenty years old, and the equipment itself is showing signs of advancing age through the increasing unreliability of its electronics. This study was begun to achieve two major objectives: i. To improve the productivity of the equipment and operating personnel, and ii. To improve the accuracy and sensitivity of the measurements by suggesting advances in the state of the art.

The findings of the study are, in summary, that there is a need for increased optical sensitivity to increase productivity, and that better design of the data collection and processing scheme can eliminate some of the unnecessary present operations. Two promising approaches to increased sensivity have been identified, conventional processing with error compensation and detection of random noise modulation. The latter of these approaches is of sufficient novelty that it is under investigation to determine its patentability.

INTRODUCTION

It is essential to have a capability for measuring the reflectance of objects in order to carry out the missions of the Space Sciences Laboratory of the Marshall Space Flight Center. Almost all objects placed in space for scientific, commercial, industrial, and military purposes must have some form of special coating to control the internal temperature. These thermal control coatings, either light colored to reflect large amounts of incident radiation or black to best radiate surplus heat into space, have organic binders and other components which are affected by the high vacuum and ultra-violet radiation characteristic of the space environment.

Furthermore, many of these space payloads have optics, either lenses or mirrors, to orient them and collect information. These surfaces are subject to contamination by the outgassing of thermal control coatings, plastic parts and electrical insulation, exhaust plumes from reaction motors, etc. Measurement of the reflectance of these items is also required to permit the evaluation of data collected and prediction of their reliable life.

Simple measurements of reflectance, reflected energy divided by incident energy, are not sufficient. For example, obtaining thermal balance for maintaining the proper internal temperature may require high reflectance in one spectral region to ward off incoming radiation while having low reflectance in another spectral region to promote the outward radiation of heat energy. Thus the reflectance must be measured as a function of wavelength over a wide spectral band. Typically, wavelengths from 200 nanometers (nm.) in the ultra-violet to 2500 nm. in the infra-red are required.

OBJECTIVES

Means currently exist for conducting such measurements. Two Beckman Model DK-2A Spectroreflectometers are in use, and a Cary of similar operating characteristics is to be equipped with auxiliary items to enable its use. These instruments are well-built, massive pieces of laboratory equipment. Unfortunately, their design is now about twenty years old, and the electronics in particular have become unreliable and noisy. Their claimed accuracy is only two per cent; this together with the noisiness can easily obscure effects sought.

Also, these instruments are not designed for high-volume ("production") use. All adjustments must be made by hand. Frequent zero-ing and setting of the maximum scale value must be carried out by hand in a typical measurement sequence. Some modifications have been carried out to allow computer control of the wavelength selection function, and the reading of the output by the computer has been arranged for after a fashion. However, these modifications have resulted in the measurements being compute-bound because the computer is time-shared with other jobs.

Thus the long term objectives of the study undertaken were twofold:

1. To improve the productivity of the equipment and operators to handle a growing demand for reflectance measurements.

2. To advance the state of the art, if possible, allowing more precise, more repeatable measurement of reflectance in keeping with the growth of instrumentation ability generally due to the switch from vacuum tubes to solid state devices and from analog to digital measurement.

Naturally, it has not been possible to achieve these objectives in nine weeks. A good start has been made toward identifying feasible alternatives for further study. A more detailed description of the problems and the feasible alternatives is presented in the next sections.

GENERAL EQUIPMENT DESCRIPTION AND USAGE

The general functional diagram of the existing equipment is shown in Fig. 1. The upper half of the figure is the path of the information in the optical portion of the instrument; the lower portion shows the electronic schematic.

The monochromator serves to select a narrow band of light wavelengths from the broad-band sources, either a tungsten filament lamp for the visible and infra-red bands or a deuterium lamp for the ultra-violet. The 480 Hz. chopper interrupts the light to provide a (relatively) high-frequency carrier for later alternating current amplification. The slits open and close under servo control to keep the amount of light energy passing to the reference and the sample constant whenever the source is rich enough to permit this constant energy mode of operation. A secondary advantage to servoing the slits is that, with narrow slits, the bandwidth is small and hence the spectral resolution is high. This allows narrow absorption bands to be seen, a very desirable means of identifying contaminants.

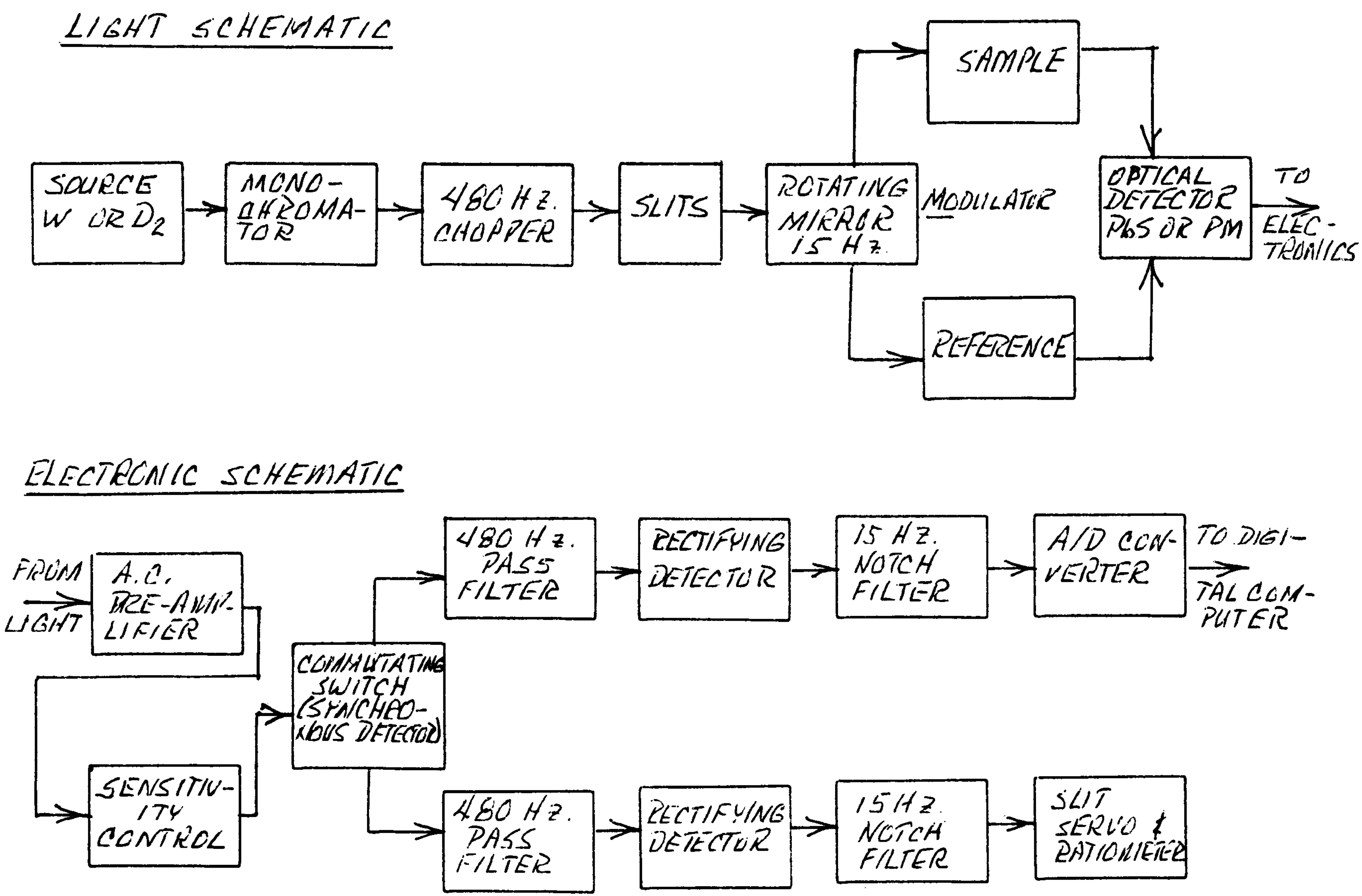

Figure 1 -- Functional diagram of present equipment

The next functional block is a rotating mirror which passes the light along two paths so that it alternates between impinging on the sample and scattering in a diffuse way on the inside of an "integrating sphere". This mirror rotates at 15 Hz., thus modulating the 480 Hz. carrier created by the chopper in a double sideband manner. At this point we now have an audio carrier modulated with a low frequency whose amplitude is the difference between the signal and the reference energies.

The integrating sphere contains the sample and detectors, and is coated on the inside with magnesium oxide to provide the highest possible reflectance. The purpose of the integrating sphere is to provide a field of view for the detectors independent of the geometry of the detectors and the sample, which it does because of the special symmetry properties of the sphere.

The optical signal is now converted into an electronic signal by one of two detectors, a lead sulphide (PbS) cell with 50 volts bias applied to it or a photo-multiplier (PM) tube with 700 volts operating through ten multiplier stages. The PM tube has a swamping resistor network selected by a switch to reduce the sensitivity by a factor of 20 when operating in the visible and near ultra-violet regions. Its photocathode is selected to maximize the response in the ultra-violet, and therefore the lead sulphide detector is the detector of choice for the visible and infra-red regions.

The 480 Hz. carrier with 15 Hz. modulation sidebands is amplified by an AC amplifier to raise the signal level to severl volts. The gain of this amplifier is adjustable to contol the amplitude of the background noise and, presumably, to prevent overloading and consequent distortion of the signal in later amplifier stages.

The next function is to demodulate (synchronous detect, demultiplex, or homodyne detect are other synonyms) the sample and reference information. This produces two signals which have been amplified by the same amplifier chain, and have therefore been subjected to the same distortions, lack of linearity, etc. Each of these 480 Hz. signals is rectified, and any remnants of the 15 Hz. modulation is removed by notch filters. The sample rectified current is proportional to the light energy returned by the sample, and the reference current is proportional to the energy returned by the perfectly reflecting magnesium oxide sphere wall.

The reference current is next used to servo the slits to keep the reference energy flux constant (if possible). It is also used to manually position the full-scale value (100%) of the built-in ratiometer. Then through a rather complex scheme using the built-in plotter pen positioner (which is not used for plotting) the sample signal adjusts a potentiometer which provides a voltage proportional to the reflected energy of the sample. This voltage feeds an analog-to-digital converter which ultimately supplies the measured voltage to the digital computer. The digital computer then stores the point pair of wavelength and response.

If the light sources were powerful enough to always provide enough light so that the slits were never wide open, the reference voltage driving the ratiometer and servoing the slits could also be recorded and the reflectance computed immediately. Unfortunately the sources run out of energy at the extremes of the measurement range. Thus the slits open further and further until they are at their maximum opening. At this time the full scale setting on the ratiometer is in error, and the energy reflected from the sample is likewise reduced, not due to low reflectance, but because of lower incident energy. This problem is overcome in the following way.

If the sample is moved aside while still in the sphere, the sample beam will no longer impinge on it but will look at the sphere wall, the same as the reference beam. Thus a run made under these conditions will record the true incident amount of light. Dividing the first (sample) run by the second (reference) run in the digital computer will produce a correct reflectance reading, corrected for the loss of energy in the sources. The price is, of course, a doubling of the time required to obtain a single sample-only reading. The system does work, and works well, but is rather inefficient because of inflexible analog instrumentation. A significant potential source of errors is the long period of time (a minimum of three minutes) between readings of the sample and the (ultimate) reference in the two-pass procedure.

OPERATIONS ANALYSIS

Beside giving attention to the equipment aspects of measuring reflectance, the operational use of the equipment was also studied. An operation process chart of the use of the DK-2A was constructed while samples from the last flight of the Induced Environmental Contamination Monitor (IECM) were being measured. The results are shown in Table I, and summarized in Table II.

The symbols in the first column of Table I are the standard charting symbols used to classify activities. The circle represents a productive operation, something which changes the value of the product or generates information. The D-shaped symbols are delays or idle time. In this case the operations are a classic example of a machine-paced job. The operator idle time is about 80% of the time required to

Table I -- Operation Process Chart of DK-2A Use

Operator	Description	Time
○	Change sample in holder	2 min.
○	Reinstall in sphere, W lamp on	1
○	Set calibration range & zero	1
D	Reference run, long wavelength	9
○	Expose sample	0
D	Sample run, long wavelength	8
○	Retract sample	0
○	Set calibration range & zero	1
D	Reference run, medium wavelength	4
○	Expose sample	0
D	Sample run, medium wavelength	4
○	Retract sample, D_2 lamp on	0
○	Set calibration range & zero	1
D	Reference run, short wavelength	3
○	Expose sample	0
D	Sample run, short wavelength	3
D	Plot, store data (if good run)	2

Table II -- Operation Process Chart Summary

Run	31 min.
Recalibrate	3
Change samples	3
Plotting	2
Total	39 min.

perform one complete cycle of the measurement job. Even worse, the idle time is broken up into small chunks of from three to eight minutes, leaving little time for other constructive work. Because technicians and co-op. students are the operators and are highly motivated people, the usual consequence is boredom and distaste for the job.

EXPERIMENTS

Fortunately, the DK-2A with the integrating sphere became available for a period of several weeks for some temporary modifications and measurements. These were carried out primarily to correct some operating deficiencies known to exist and to conduct measurements to provide design constraints for redesign of the electronics.

A relay rack with an oscilloscope and NIM-bins was also available. The NIM-bins are electronic packages designed to be modular elements of nuclear measuring systems. Modules were available in the form of AC broad-band amplifiers (1 Hz.- 100 KHz.), selective amplifiers with Q's up to 100 over the same frequency band, and "lock-in" amplifiers, which are synchronous detectors but may also be used as simple r.m.s. (root-mean-square) AC voltmeters. The theory of the use of these elements will be presented later in connection with the development of a model of modulation and the signal-to-noise ratio of the optical measurements.

EXPERIMENT 1 -- In the first experiment the electronics of the DK-2A were bypassed entirely. Using high-impedance probes to avoid loading any existing circuits, connections were made to the output of the detectors at the wiper of the selector switch. Thus the outputs of the detectors could selectively be applied to the input of the oscilloscope and the NIM-bin amplifiers. For this experiment, only voltages were measured using the calibrated input of the oscilloscope. The following results were obtained:

> 480 Hz. amplitude on long wavelength band
> ~1.5 mv. min., 3.5 mv. max.
>
> The noise (non-square wave portion) at the
> 15 Hz. modulation frequency ~1 mv.

Thus the minimum detectable difference at the claimed accuracy of 2% is not more than

$$(0.02)*(1.5\ \text{mv.}) = 30\ \text{microvolts.}$$

A complete series of measurements across the entire spectrum was taken, showing the maximum voltage to be 310 mv. at 500 and 600 nm. wavelength using the PM tube in its reduced sensitivity (X1) mode. The light output seemed to be very small using the deuterium (D_2) lamp. The reason for this has not been determined; the source has been changed without noticeable difference.

Other things noticed were:

> Shielded probes with short ground leads are necessary to prevent the pick-up of considerable computer generated "hash".
>
> The use of an internal 10 KHz. low-pass filter in the scope eliminated a lot more hash in the detector output.
>
> There was a large 60 Hz. (power line frequency) component in the signals, as well as a large 180 Hz. (third harmonic) component.

At this point a rather seredipitous observation was made. With very low light levels, the current pulses due to the collection of individual photons by the photocathode could be observed on the scope. Individual pulses could still be seen even at high light levels. A few moments' reflection showed that this so-called "shot noise" is composed of rare events; rare events follow a Poisson probability distribution; the standard deviation (root-mean-square AC component) of a Poisson distribution is the square root of the mean (DC component); therefore the shot-noise AC component is related to (correlated with) the DC component, which we wish to measure. Experiment 2 was set up the test a practical way of implementing this technique.

EXPERIMENT 2 -- Just measuring the AC component of the signal would not be a practical method because of the large amounts of computer "hash", power line frequencies and harmonics, and other noise present. A suitably chosen narrow bandwidth would, however, contain noise energy proportional to the shot-noise energy because the individual impulses of which it is composed each have a uniform power spectrum, while avoiding the spurious low frequency content.

The equipment available was set up to accomplish observation of this scheme. The arrangement is shown in Fig. 2. The selective amplifier was set to give a Q of 100 and was tuned from 2.5 KHz. to 10 KHz. Best results were obtained at about 6 KHz. The lock-in voltmeter was operated in its rms. voltmeter mode. While no quantitative measurements were made at this time, the system was sensitive enough to detect the leakage into the integrating sphere of a flashlight played on the darkened room's walls. Similarly, shot noise energy was detected by the PM tube from both the tungsten and deuterium sources across the

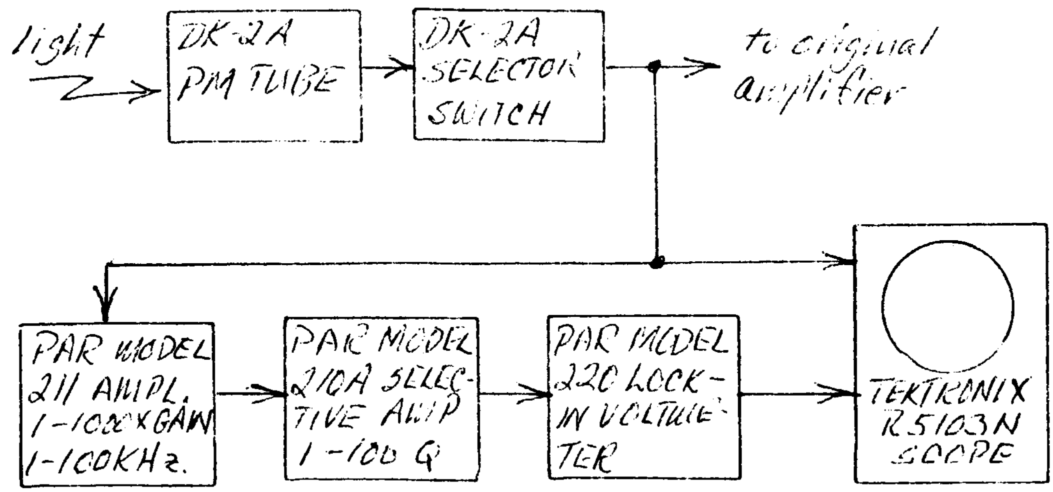

Figure 2 -- Shot noise energy set-up

entire optical spectrum, although this could be spurious radiation not trapped by the monochromator.

A theoretical analysis was conducted to find the form of the response curve and evaluate the sensitivity of the method. Horowitz and Hill,[1] p. 288-9, give the shot noise due to a DC current as

$$I_{rms} = (2qI_{DC}B)^{1/2} \quad \text{(IX-1)}$$

where q is the electron charge, $1.6(10)^{-19}$ coulombs, I_{DC} is the DC current, and B is the bandwidth. The ratio of two currents will vary as the square-root of the ratio of the bandwidths over which they are measured:

$$I_1/I_2 = (B_1/B_2)^{1/2} \quad \text{(IX-2)}$$

A 1 microA. current has a 10 KHz. bandwidth rms. noise current of

$$[2*1.6(10)^{-19}*(10)^{-6}*(10)^{4}]^{1/2} = 57 \text{ pA.} \quad \text{(IX-3)}$$

Across a 1 Mohm load resistor this yields an rms. shot noise voltage of 57 microV. Finally, if the bandwidth is reduced by a resonant filter of Q = 100, the shot noise current is reduced by the narrower bandwidth by a factor of 1/10; but the resonant impedance is 100 times higher, yielding a net gain of 10 for a total rms. noise voltage of 570 microV.

This is easily measured. Again, quantitative measurements were not attempted beyond measuring the peaks of the individual pulses as 100 microV. across a 100 Kohm load.

The method does have two significant advantages which recommend further investigation:

1. The high selectivity permitted and the freedom to locate the center of the narrow band over a relatively wide frequency range give promise of relative immunity to other sources of noise.

2. The square-root relation between rms. noise voltage and average DC voltage means that the dynamic range of the amplifiers can be smaller.

The ideas brought out in Experiment 2 have sufficient novelty that they are under investigation for patentability.

EXPERIMENT 3 -- In the third experiment, the objective was to learn about the sensitivity and nonlinearity of the detectors so that a new electronics package could be designed. To this end the experiment was designed to use the NIM-bins to allow the collection of data independent of the old electronics.

Three different conditions were established to use all the usual combinations of sources and detectors. Since we were interested in finding nonlinearities, manual control of the slit size was elected, and the slits varied from 0.04 mm. to 2 mm. Since the energy varies as the square of the linear slit dimension with the sources used, the energy ratio was 2500 to 1. The lower limit occurred when the source did not have enough energy to be detectable, usually occurring at 0.04 mm.

It was found that at the larger slit openings the amount of energy available was too great for the instrumentation to operate at unity gain without overloading. A three-decade attenuator, matched to the impedances of the detectors and the amplifier, was constructed and used. A modification to the DK-2A was performed to allow the 480 Hz. chopper to be observed via a separate channel. This chopper signal was used to synchronize the synchronous detector in the lock-in amplifier. The general configuration of the equipment is shown in Fig. 3.

The modulation theory used here is thoroughly covered by any modern text in Communication Theory. See, for

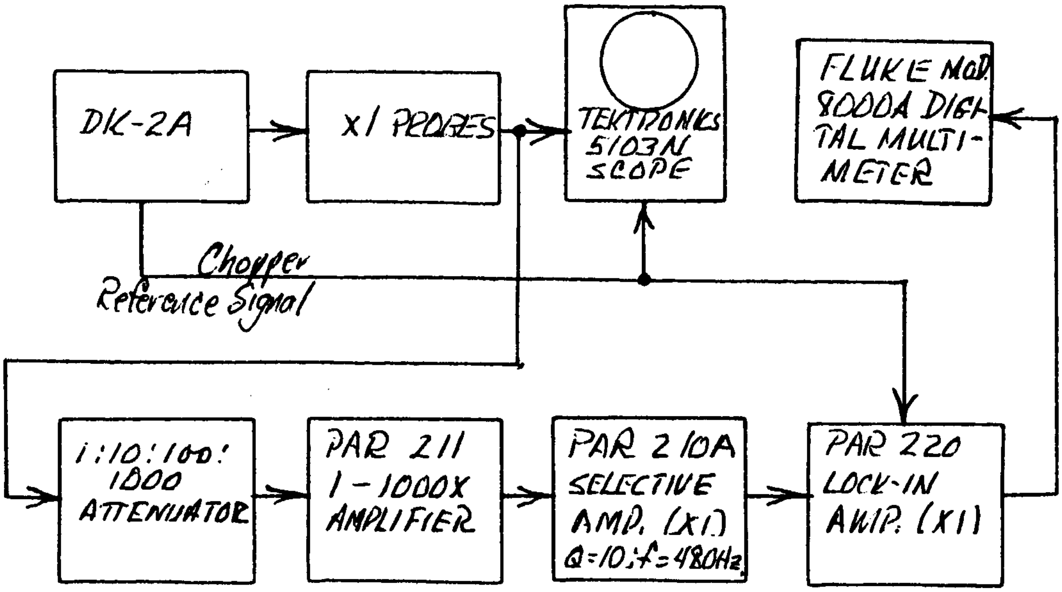

Figure 3 -- Equipment arrangement for Experiment 3

example, Gregg[2]. We represent the modulating signal by m(t). In our case this is the voltage representing the light energy incident on the detector. A carrier, c(t), the 480 Hz. chopper square wave, produces double sidebands centered around the carrier frequency. The resultant is s(t). After transmission through the communication channel, here the light detector and amplifiers, the signal, now called r(t) is demodulated by some means. The result, m'(t), is a representation of the original which is more or less faithful depending on the nonlinearities and noise encountered in the detectors and amplifiers:

$m'(t) = m(t)$	Perfect rendition
$= m(t) + n(t)$	Additive noise
$= a(t)*m(t)$	Multiplicative noise (not common in non-radio communication)
$= f(m(t)) + n(t)$	Nonlinearities and additive noise

The last two of these are usually avoidable in a well-designed information system. The first one is only an ideal, leaving us to deal with the second form, additive noise.

A full treatment of noise figure and signal to noise ratios is beyond this report (but should ultimately be done). A briefer, ad hoc derivation of an important relation affecting this study is now given. Consider the ideal optical signal waveform shown in Fig. 4. When the chopper obscures the light beam there is no output except that due to the noise term of the additive noise form above. This has been sketched in Fig. 4 as the wavy line in the two segments of the chopper square wave. The upper horizontal line represents either the true sample reflected energy, S, or the reference reflected energy, R, depending on the position of the sample/reference deflecting mirror in the

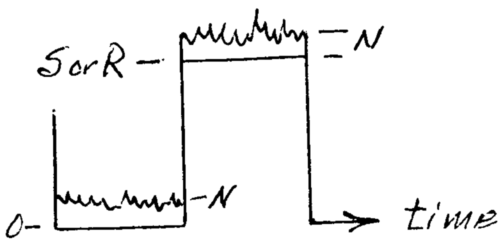

Figure 4 -- Signal showing effect of additive noise

DK-2A. What we wish to measure is the true value of the ratio S/R, but we actually measure (S + N)/(R + N). Is there any way of obtaining the former from the latter? Obviously, if we knew the value of N, the instrumental noise, we could subtract it out of each term of the measurement and get the true S/R. After some algebraic manipulation we can obtain

$$S/R = [(S + N) - N]/[(R + N) + 1]. \qquad \text{(IX-4)}$$

Although this still doesn't look too hopeful, the terms in parentheses are the measured quantities, and the term on the left is the desired ratio.

Suppose now that we take several measurements on the same sample with the same source, detector, and wavelength combination. Equation 4 can be rearranged, and subscripts added to index measurements and unique measurement errors:

$$(S + N)_i = (S/R)*[(R + N)_i + 1] + N + (\text{unique error})_i \qquad \text{(IX-5)}$$

This has the linear form

$$y = a + b*x + (\text{unique error}) \qquad \text{(IX-6)}$$

which is a linear regression! So repeated measurements such as those taken in Experiment 3 with varying slit widths can be used to estimate both the true reflectance, S/R, and the instrumental noise, N, as long as the signal channel remains linear. The estimators are

$$\begin{aligned} N &= a \\ S/R &= b \\ (R + N)_i + 1 &= x_i \\ (S + N)_i &= y_i \end{aligned} \qquad \text{(IX-7)}$$

<u>Note carefully the need to add one to each value of the reference reflectance</u> in calculating the regression. This term can be interpreted as arising from having normalized all measurements by the amplitude of the noise.

A similar analysis shows a similar form for errors introduced by phase errors in the synchronizing signal (from the chopper) and by commutation errors caused by lack of symmetry of the open and closed segments of the chopper. They will all contribute to the noise term, N, above. However the signs of these two errors are opposite from that of instrumental noise! This presents some interesting possibilities for cancelling instrumental noise effects by the intentional introduction of phase or commutation errors in the synchronous detection.

The technique derived above was used to fit the data of Experiment 3, with the expectation that the noise term, N, would be of the order of a few microvolts. The results were irregular and disappointing, however. Account was carefully taken of the attenuator setting and the gains of the various amplifiers in the signal amplifiers. After considerable trial and error, it was finally discovered that the experimental measurements only fell into an orderly array when the gain and attenuator settings were ignored! Table III summarizes these results.

Table III -- Experiment 3 Regression Results

Source	Detect.	Atten.	Gain	# Pts.	N Est.	%R Est.	Coeff. of Determination
W	PbS	1/10	10	9	0.888mv.	89.2	0.999,991
(1200nm.)			100	7	0.828	88.4	0.999,910
			1000	9	0.753	91.1	0.999,860
W	PMx	1/1000	1	9	1.194	104.3	0.997,279
(575nm.)	20		100	6	0.842	94.3	0.999,757
D	PMx	1/10	1	6	0.920	94.2	0.999,994
(575nm.)	20		10	4	0.959	95.7	0.999,992
			100	5	0.967	97.1	0.999,986

The table displays several interesting results. First, the noise estimates approximate one millivolt on an instrument set to read ten millivolts full scale. As already noted, the gain and attenuator settings were disregarded in these calculations, indicating that the 1 mv. noise is being generated in the final stages of the instrumentation. That the results are no fluke is indicated by the fact that the coefficient of determination (square of the correlation coefficient) is very high for most cases. One minus this quantity measures the amount of variation in the dependent variable <u>not</u> explained by the regression equation.

One can only interpret these results as suggesting the instrumentation, probably the lock-in voltmeter, has sufficient internal noise in the synchronous detection process that a noise magnitude approximating 9% of full scale exists.

The table does show that there is some apparent nonlinearity in the detector response, because the corrected values of the reflectance do not remain constant at different incident energy and gain settings. In one case, an impossible result of 104% reflectance was indicated. It is also possible that the lack of agreement is due to the originally anticipated detector noise phenomenon. Pulling this effect out of the data would require a multiple regression analysis; this there was insufficient time to do.

RESULTS AND CONCLUSIONS

In summary, the following results have been obtained:

1. Nonlinearity of the detectors is indicated.

2. Instrumental noise currently limits the investigation.

3. A method of correcting readings for noise and errors, with application to digital computation and control, has been developed.

4. A novel method of measurement based on shot noise modulation has been discovered.

5. A microprocessor-based digital computer has been assembled and made operational for dedicated use in recording, computation, and control of the reflectance measurements.

The following conclusions have been reached:

1. The original study objectives are still valid and achievable.

2. There is a need to locate and eliminate the source of the instrumental noise.

3. Failing this, a theoretical noise study is needed to determine the ultimate capability of the synchronous detection method.

4. Some effort should be expended to further explore the novel noise modulation measurement method.

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