

Functional Enhancements in Used Oil Analysis Spectrometers

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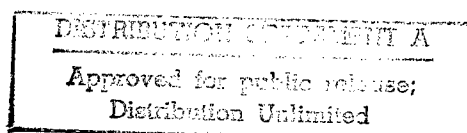
Abstract: Spark emission spectrometers using the rotating disk electrode (RDE) technique have become the workhorses and primary analytical tool of most machine condition monitoring programs based on oil analysis. This paper describes several new developments that have put new life into this established and well accepted used oil analysis technique. They include performance enhancements, automation and additional capabilities.

Digital technology has been applied in the design of excitation sources, optical and readout systems to greatly improve stability, maintainability and capabilities. A robotics system has also been developed to provide automated and unattended sample introduction and analysis. Several recent new inventions and developments have also augmented the applications and capabilities of the RDE technique. They include, 1.) the ability to analyze large wear particles, 2.) a modification to include the ability to analyze diesel engine coolants and water, 3.) the development of an additional small optical system to make it possible to analyze sulfur in oil and fuel and 4.) a modification to analyze the conductivity of a used oil sample in the same step as the normal wear metal analysis.

Key Words: Oil analysis spectrometers, rotating disk electrode (RDE) spectrometer, automation, solid state excitation, large particle size analysis, coolant analysis, conductivity analysis, sulfur analysis.

Introduction: Oil analysis spectrometers have been in use for the analysis of wear metals, contaminants and additives in lubricating oils for almost 50 years. They have become the mainstay and primary analytical tool of most machine condition monitoring programs based on oil analysis. Spectrometers have evolved from large instruments that take up the better part of a laboratory, to smaller table top instruments. Analysis times have decreased from hours to seconds, and the instruments no longer have to be operated by experts to obtain excellent analytical results.

Spectrometers using the rotating disk electrode (RDE) technique, long an established and accepted method, had become somewhat forgotten and ignored due to the commercialization of the more modern inductively coupled plasma (ICP) excitation technique. In the last five years, all this has changed. There has been what some consider to be a "rediscovery" of the RDE oil analysis technique due to a variety of innovations, inventions and expanded capabilities, all of which have lead to more



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effective and productive used oil analysis programs in the military and commercial sectors.

Digital Technology Enhancements: Digital technology has had a major impact in the design and capabilities of oil analysis spectrometers. It has influenced the design and features of excitation sources, optical system size and capabilities, and readout and data management systems.

All excitation sources are based on simple electronic circuitry using discrete components. Development trends have been towards solid state excitation circuits which result in cost, size and serviceability improvements. One of the side benefits of applying solid state technology is the ability to eliminate transformers that are bulky and extremely heavy. Excitation sources for RDE spectrometers with solid state ignition circuitry have recently been introduced to the market. They improve instrument stability and greatly reduce operator periodic maintenance requirements. The need to adjust source frequency to compensate for voltage or environmental changes has also been virtually eliminated. This is one of the few areas where digital technology actually slightly increases the cost, but the advantages in performance and reliability are well worth it. Additional advances in solid state circuitry can be expected in the next few years.

The optical system of a spectrometer has always been the primary factor in determining the size of an instrument. Improvements in gratings have made it possible to reduce size without sacrificing performance or dispersion. The early instruments with 3 meter (9 feet) focal curves were replaced with 2 meter, then 1 meter and today as small as 0.3 meter optics. Reduction in focal curve size due to solid state detectors have made it possible to build much smaller instruments with better stability. Advancements in fiber optic technology have also led to further improvements in stability, flexibility and size of optical systems.

The optical system is still the most expensive, the largest and the most delicate component of an instrument. However, replacements for expensive photomultiplier tubes in the form of solid state charged coupled devices or CCD detectors has changed all this. This technology provides devices, by a variety of processes, consisting of many small light sensitive semiconductors (pixels) which may be arranged in a single row (linear array) or as a rectangle or square (area array). CCD technology is widely applied in such commonplace and reliable devices as video cameras, copy and fax machines. Consequently, a robust industry exists to supply and improve such devices and they are available at favorable prices, Fig. 1.

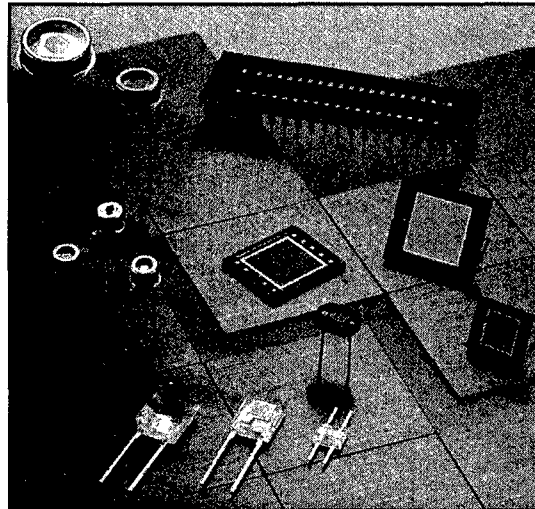


Figure 1, Examples of CCD Detectors

It is now possible to create smaller and more capable optical systems by placing CCD arrays along the focal surface of spectrometer optics, Fig. 2. This eliminates the need for expensive photomultiplier tubes and selection of elements prior to delivery of an instrument to a customer. CCD technology makes virtually any wavelength available for an analytical program and can be selected through the operating software. Resolution may not be improved in all cases, but accuracy can be improved since CCD arrays offer an unlimited possibility of selecting primary wavelengths and locations for background subtraction and referencing, with much greater freedom than current photomultiplier tube detector technology allows.

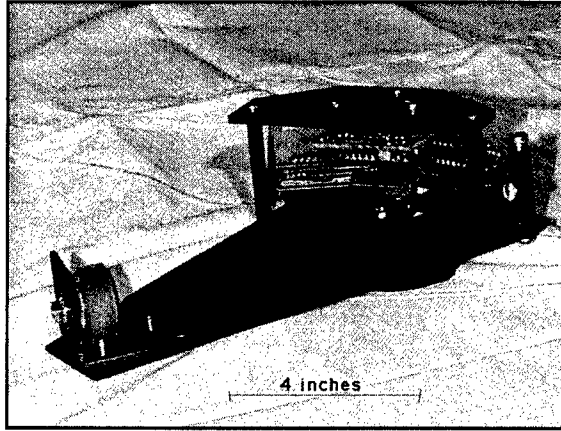


Figure 2, Optical System with CCD Detectors

CCD technology has already been incorporated in several commercial ICP spectrometers, but not demonstrably with the cost/benefit that it might ultimately derive. It is, however, an area where we can expect to see major developments in the next year or two.

Digital technology was applied in spectrometer readout systems with the introduction of the early mini-computers. Computerization made it possible to rapidly reduce vast amounts of data into figures and units readily understandable by the user. In the past, readout system hardware was complex and differed greatly among manufacturers. Today, except for one or two circuit boards, the personal computer is becoming more and more the only electronic readout system required for the spectrometer. It provides instrument control, data processing and troubleshooting.

Automation: Automation for the RDE spectrometer has always been difficult due to the need to replenish the graphite electrodes after each analysis. The rod electrode, in particular, has been a challenge to handle by robotics, since it not only must be sharpened after each burn, but also becomes shorter after each sharpening. A novel new robotics system has been developed which overcomes these hurdles.

The practical solution to RDE spectrometer automation is to use two graphite disc electrodes, Fig. 3. This eliminates the need to sharpen electrodes and greatly simplifies operation. The automation system consists of two parts, a robot to exchange the consumable disk electrodes and an automatic sample changer. The robot dispenses new graphite electrodes for each analysis and removes the used ones. The need to set the analytical gap size has also been eliminated since the disk electrode shafts form a fixed gap. An robotics arm in the sample changer automatically introduces each of 48 oil samples in succession, at a rate of 80 samples per hour, and without the need for sample dilution.

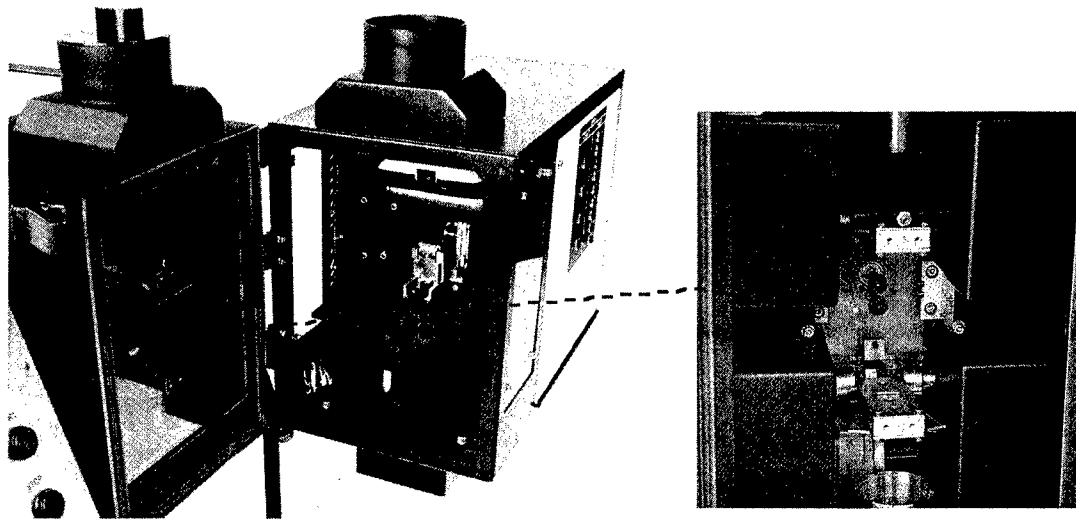


Figure 3, Robotics for RDE spectrometer

The entire automation system mounts to the spectrometer sample stand and fulfills all the functions of sequentially introducing and removing oil samples and exchanging graphite electrodes. It is self contained and works independently of the spectrometer operating software. Although operation is automatic, it also has the capability to manually sequence through each of the robotics functions. Automation also improves repeatability by eliminating operator variances and maintains JOAP correlation.

Large Particle Size Analysis Capability: The ability to detect and quantify large wear and contaminant particles has always been considered to be one of the shortcomings of spectrometers. Few will agree as to the actual detrimental impact on a condition monitoring program, but most will agree that the practical particle size limitation of spectrometers are at particles below 5 micrometers for ICP and AAS, and at particles below 10 micrometers for RDE [1, 2]. Today, the particle size limitation of RDE spectrometers has been eliminated with simple ancillary systems such as the rotrode filter spectroscopy (RFS) method.

Rotrode filter spectroscopy (RFS) makes use of the fact that the carbon disc electrodes used in rotating disc electrode (RDE) spectrometers are themselves porous. A fixture is used to clamp the discs so that oil can be drawn through the outer circumference of the discs when a vacuum is applied to the inside of the discs, Fig. 4. The particles in the oil are captured by the disc. The oil is then washed away with solvent, the disc is allowed to dry, and the particles are left on the disc electrode so that they are vaporized and detected when run on the RDE spectrometer. It is a technique whereby the normal analysis of the oil sample serves to provide data on particles that are dissolved to 10 micrometer in size, and the RFS technique analysis provides data on large particles [3]. A multi-station fixture is used so that a number of samples can be filtered at one time. Several commercial laboratories offer RFS to provide a more comprehensive analysis of used oil samples.

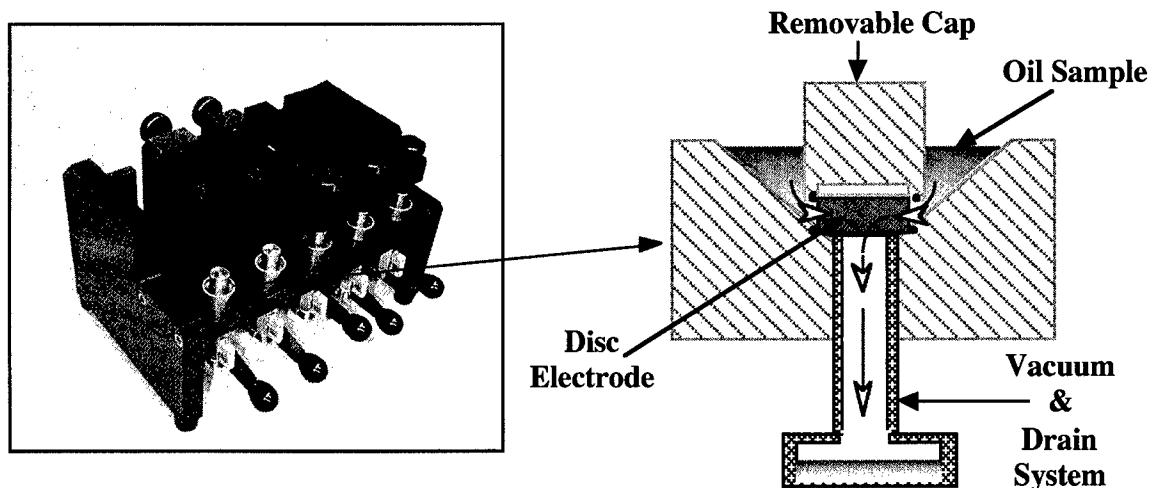


Figure 4, Rotrode Filter Spectroscopy (RFS) Fixture

Expanded Applications Capabilities: Although the RDE spectrometer is still designed primarily for used oil and fuel analysis, several methods and recent enhancements have increased productivity through expanded capabilities. They include the ability to analyze engine coolants, sulfur in oil and fuel, and oil degradation through changes in conductivity.

A used coolant analysis program determines both the coolant condition and the presence of any contaminants or debris. The coolant fluid can be used as a diagnostic medium as the coolant carries not only heat away from the engine parts but also carries fine debris from the interior surfaces of the cooling system. Analysis of the wear debris can provide important information about the condition of the internal parts of the cooling system.

Some machine condition monitoring programs have gone beyond used oil analysis and also provide data on the coolant system. The application of coolant analysis, however, has been limited due to additional cost and the time required to analyze samples. Until recently, ICP or AAS spectrometers have been used exclusively for this purpose. Although they provide good analytical data, the ICP is expensive and complicated to operate, and the AAS is slow since only one element is analyzed at a time. Today, several major commercial oil analysis laboratories have switched to the RDE technique for coolant analysis. This was made possible with minor hardware and software modification to the RDE spectrometer. It has been shown that the RDE technique correlates well with ICP and AAS techniques on new coolants and is more efficient on used coolants that contain particulates [4].

On-site sulfur analysis in lubricating oils and fuels has long been desired, but until recently, was impossible with RDE spectrometers. When a sample is sparked, the light emitted by sulfur is of such short wavelength that it does not reach the optics because it is absorbed by oxygen in air. ICP spectrometers with optics housed in a vacuum system had to be used for this purpose. This is no longer necessary as RDE spectrometers can be modified to include the capability to analyze sulfur. A compact, argon purged optic mounted near the sample stand of the spectrometer has been adopted for this purpose, Fig. 5. It is argon purged but consumption is minimal due to

the small size of the optic and the short optical path to the spark. It can be turned on and off as required to coincide with those samples that require sulfur analysis. With this enhancement, sulfur can be analyzed on the RDE spectrometer along with the remaining routine elements.

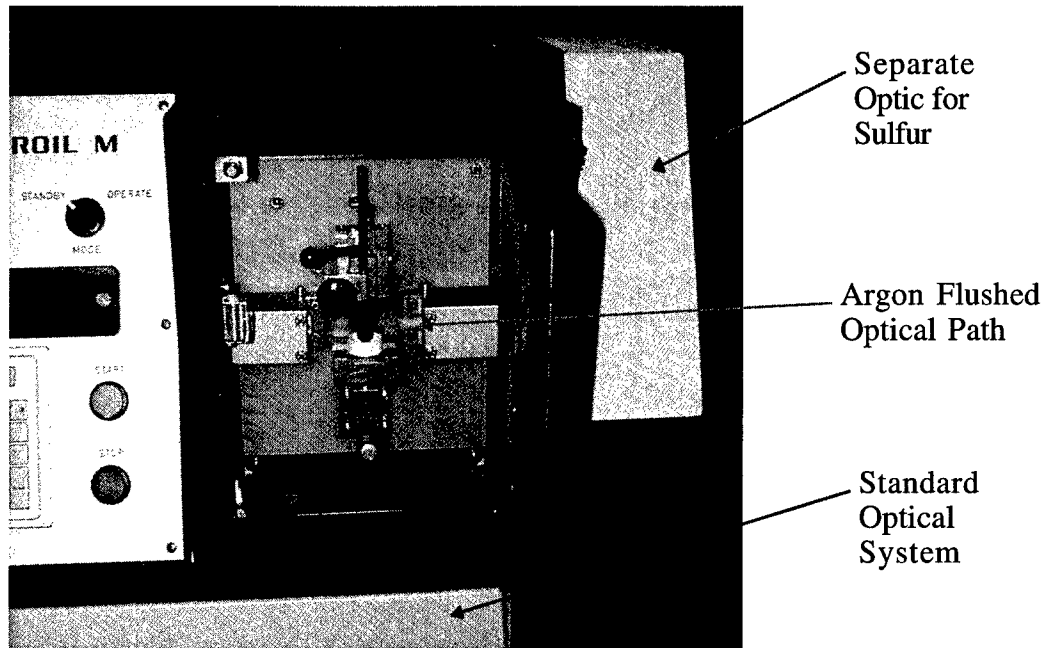


Figure 5, Sulfur Analysis Optical System Mounted on RDE Spectrometer

Finally, another RDE enhancement to analyze oil degradation along with routine wear metals is currently under test by the U.S. Air Force at Wright Laboratories. The technique is referred to as tandem conductivity technique (TCT). It measures used oil's conductivity, a sign of degradation and an indication of "burnt oil". A RDE spectrometer has been upgraded with TCT which includes a measurement circuit, sample table modification to adapt a special oil sample vessel, and software for calibration and readout of the conductivity measurement, Fig. 6.

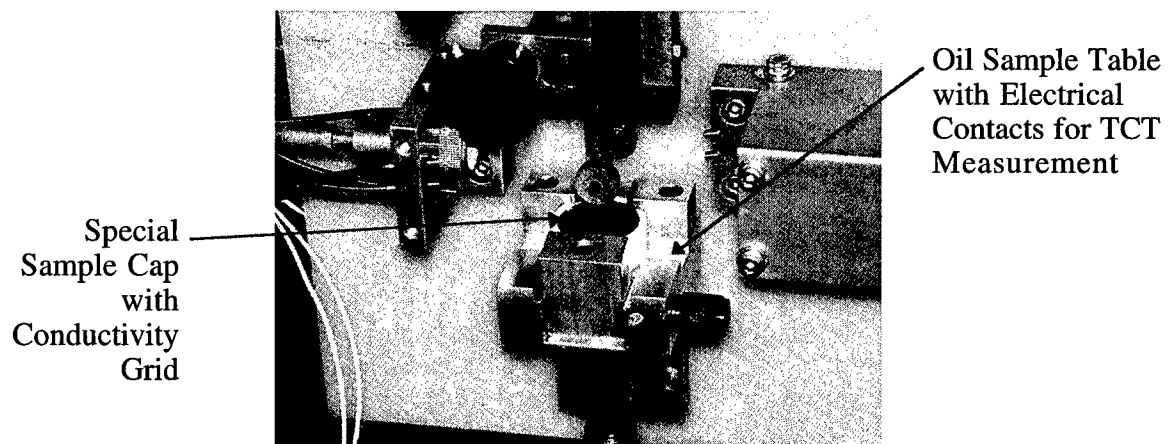


Figure 6, TCT Sample Stand

The RDE analysis with TCT is almost identical to current procedures and the conductivity measurement is made during the first four (4) seconds of analysis followed by the normal 30 seconds of wear metal analysis. Initial conductivity testing at the JOAP TSC in Pensacola, FL on actual "burnt oil" samples has been encouraging and has shown good correlation to other methods [5].

Conclusion: The rotating disc electrode (RDE) spectrometer has had a long and successful history as a workhorse instrument in the oil analysis laboratory. It has made a major comeback in recent years; just as new techniques such as ICP were thought to be an ideal replacement. Through the application of new technology, innovative inventions and common sense, the RDE spectrometer has re-established itself as the primary oil analysis spectrometer in military and commercial machine condition monitoring programs.

Spectrometers for oil analysis have evolved from instruments that had to be used in environmentally controlled laboratories and by expert and well trained scientific personnel. Today they have become easier to operate, smaller in size, mobile, and more robust for use in non-laboratory environments. They have become more efficient due to automation and expanded capabilities beyond routine oil analysis. The net effect has been an analytical capability that provides a great amount of information immediately and at the site where it is actually needed.

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