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Y-12

OAK RIDGE Y-12 PLANT

LOCKHEED MARTIN

Final CRADA Report for CRADA Number Y-1295-0385

SURFACE INSPECTION MACHINE INFRARED (SIMIR)

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ABSTRACT

This Cooperative Research and Development Agreement was a one year effort to make the surface inspection machine based on diffuse reflectance infrared spectroscopy (Surface Inspection Machine-Infrared, SIMIR), being developed by Surface Optics Corporation, perform to its highest potential as a practical, portable surface inspection machine. The design function of the SIMIR is to inspect metal surfaces for cleanliness (stains). The system is also capable of evaluating graphite-resin systems for cure and heat damage, and for measuring the effects of moisture exposure on lithium hydride, corrosion on uranium metal, and the constituents of and contamination on wood, paper, and fabrics. Over the period of the CRADA, extensive experience with the use of the SIMIR for surface cleanliness measurements have been achieved through collaborations with NASA and the Army. The SIMIR was made available to the AMTEX CRADA for Finish on Yarn where it made an very significant contribution. The SIMIR was the foundation of a Forest Products CRADA that was developed over the time interval of this CRADA. Surface Optics Corporation and the SIMIR have been introduced to the chemical spectroscopy on-line analysis market and have made staffing additions and arrangements for international marketing of the SIMIR as an on-line surface inspection device. LMES has been introduced to a wide range of aerospace applications, the research and fabrication skills of Surface Optics Corporation, has gained extensive experience in the areas of surface cleanliness from collaborations with NASA and the Army, and an extensive introduction to the textile and forest products industries. The SIMIR, marketed as the SOC-400, has filled an important new technology need in the DOE-DP Enhanced Surveillance Program with instruments delivered to or on order by LMES, LANL, LLNL, and Pantex, where extensive collaborations are underway to implement and improve this technology.

BACKGROUND

The application of Fourier transform infrared spectroscopy (FTIRS) to the analysis of surfaces and gases has long been recognized as a key to the solution of many Department of Energy Defense Programs (DOE-DP) development, certification, and surveillance problems. With the establishment of an FTIRS capability at the Oak Ridge Y-12 Plant, a collaborative relationship with Harrick Scientific, Inc., Ossining, New York was established to development these technologies for DOE-DP purposes. This collaboration led to the development of evacuable cells for surface analysis by diffuse reflectance[1] (IR-100 Award 1984) and remote sensing capabilities for surface inspection[2, 3] (R&D-100 Award 1989), primarily to measure the product of moisture corrosion LiOH on LiH. The diversification of this surface inspection method to areas such as the oxidation of uranium, coal and graphite-resin systems, as well as, stains on metals, woods, papers, and fabrics led to the development of a prototype portable system based on a MIDAC FTIR and a Harrick Barrel Ellipse diffuse reflectance accessory. The first of these systems was built for a NASA contractor for surface cleanliness measurements (1990) and the second was built for the Oak Ridge Y-12 Plant (1992) with the support of Lawrence Livermore National Laboratory (LLNL). This technology was used to demonstrate its applicability to inspecting LiH for LiOH in an assembly dry room at the Oak Ridge Y-12 Plant (Attachment 1), field inspection of a military aircraft for heat damage to a graphite-resin component in a military aviation depot[4], and to inspecting certain weapons parts at Los Alamos National Laboratory (LANL) for corrosion and trace amounts of organic materials such as adhesives, plasticizers, and high explosives.

Collaborations with the Surface Contamination Analysis Technology Team (SCATT), a NASA/MFSC (Marshal Space Flight Center, AL) contractors' group, led to demonstrations that this technology, with certain realistically achievable performance improvements, would significantly improve their ability to certify the cleanliness of the sand-blasted steel inner surface of man-approved solid rocket motor casings prior to bonding the fuel insulator to the casing.[5] The PI of this CRADA prepared for NASA a specification for the acquisition of such an inspection device to meet their requirements (Attachment 2) and NASA put this specification out for bids. Surface Optics Corporation (SOC, San Diego, CA) was chosen by NASA to build this instrument, and after consultation with the PI of this CRADA and evaluation of the Y-12 prototype committed to build this instrument for NASA early in calendar year 1995 for delivery in that year.

Surface Optics Corporation was a small business that specialized in research and development activities in the area of the interaction of light with surfaces, particularly those interactions that are categorized as light scattering. SOC manufactures for sale specialty optical devices such as directional reflectometers, bi-directional reflectometers, and scanning imaging spectroradiometers for use in both the visible and the infrared spectral regions. SOC was led by Dr. J. T. Neu, who had extensive experience in the aerospace industry characterizing the surface finish of aircraft and spacecraft. SOC had all the capabilities for design and manufacture of the SIMIR except for experience with the chemical interpretation of the SIMIR measurements and the related private sector market. This made partnership with LMES a very good match.
Proposal preparation for a small business CRADA between SOC and LMES Y-12 began with

this commitment to the NASA contract to assure that the resulting product performed up to the specification. Over CY 95, SOC designed and built two SIMIR instruments, designated the SOC-400, in consultation with LMES Y-12 . The design[6] was based on a MIDAC FTIR spectrometer and a Harrick Barrel Ellipse diffuse reflectance optical system all repackaged to meet the small size and weight criteria of the NASA contract. The detector preamplifier was significantly improved and miniaturized to meet the high signal-to-noise requirements. Heat transfer designed to dissipate the electrical power in such a small package was also a very important feature.

During this same time period, LMES Y-12 had supported the U. S. Army Environmental Center (AEC), APG, Maryland, in their efforts to upgrade cleaning facilities at the Corpus Christi Army Depot (CCAD), Corpus Christi, Texas, and AEC expressed interest in surface inspection for cleanliness as an additional capability for their new cleaning facilities. The Army purchased the first prototype of the SOC Surface Inspection Machine-Infrared (SIMIR) for use at LMES Y-12 to evaluate and eventually deliver to CCAD. This provided the opportunity for LMES Y-12 to debug the first SOC prototype SIMIR in time to make a number of simple, but important, improvements before delivery of the second SOC-400 to NASA.

OBJECTIVES

This Cooperative Research and Development Agreement was a one year effort to make the surface inspection machine based on diffuse reflectance infrared spectroscopy (Surface Inspection Machine-Infrared, SIMIR), developed by Surface Optics Corporation, perform to its highest potential as a practical, portable surface inspection machine. The design function of the SIMIR is to inspect metal surfaces for cleanliness (stains). The system is also capable of evaluating graphite-resin systems for cure and heat damage, and for measuring the effects of moisture exposure on lithium hydride, oxidation of uranium metal, and characterization of paper, wood, and fabrics. This CRADA effort is to assure that the production model SIMIR performs up to its potential as a surface inspection device and that the surface inspection technology developed by LMES (i.e., the ability to qualitatively and quantitatively evaluate a surface for type and quantity of oil, oxidative degradation, or sorbed moisture and hydrolysis products) is transferred to the SIMIR as a viable commercial product. This CRADA effort is also to find applications in the private sector that will lead to a stable market so that the SIMIR is available for DOE-DP purposes as an off-the-shelf item.

The above objectives have been successfully met since the SOC-400 Surface Inspection Machine-Infrared (SIMIR) has been established as a viable product in the international market place and as an essential tool in a wide range of DOE-DP Enhanced Surveillance activities at LMES, LLNL, LASL, and Pantex. Commercial applications in the aerospace industry, textile industry, forest products industry, and other manufacturing industries have been found to stabilize the SOC-400's availability.

BENEFITS TO DOE

The SIMIR, marketed as the SOC-400, has filled an important new technology need in the DOE-DP Enhanced Surveillance Program with instruments delivered to or on order by LMES, LANL, LLNL, and Pantex where extensive collaborations are underway to implement and improve this technology. The SOC-400 supplied to LMES by SOC as part of this CRADA played a vital role in decisions concerning the practicality and applicability of this technology to Enhanced Surveillance since available and demonstrable capability was required. This CRADA SOC-400 SIMIR played a vital role in the AMTEX CRADA for Finish on Yarn and formed the foundation for the establishment of the Forest Products CRADA in collaboration with Sandia National Laboratory.

GENERAL PROJECT DESCRIPTION

The approach to this project was to carry out experiments at LMES to evaluate and demonstrate the performance of the SIMIR using an instrument provided to LMES by SOC. These experiments were carried out in the context of existing collaborations with the U. S. Army and NASA, participation in the AMTEX CRADA and the Forest Products CRADA, interactions with potential sales representatives, and in the establishment of infrared surface analysis elements in the DOE-DP Enhanced Surveillance Program. Work on the other CRADAs and for DOE-DP programs were funded by those programs, but used the SOC-400 that was part of this CRADA. Design changes and modifications were fed back to SOC where they were implemented. Potential market areas were also identified. Quality assurance issues related to a consistent product were also to be addressed. The SIMIR was demonstrated to potential markets through papers published, papers presented at technical meetings, exposition booths, and visits to potential users.

This CRADA came into effect with LMES in possession of the prototype SIMIR purchased for the U. S. Army. With their permission, this SIMIR became part of the PITTCON'96 (Chicago, IL, March, 1996) Booth of LMES Oak Ridge Centers for Manufacturing Technology as a demonstration of the Oak Ridge Technology Transfer Program. A paper was presented introducing the SOC-400 SIMIR and demonstrating its diverse capabilities (Attachment 3) SOC provided brochures describing the design and function of this product (Attachment 4). This exercise produced some potential user interest, but created real excitement in the community of FTIR sales representatives. Among the interested parties were MIDAC sales representatives and the ANADIS Instruments that represents MIDAC and Harrick Scientific in The Netherlands.

The LMES PI traveled to San Diego, CA with the prototype SIMIR belonging to the U. S. Army to SOC (March, 1996) to participate in its upgrade to the status of the SOC-400 delivered to NASA (November, 1995), and to critique the status of the SOC-400 SIMIR. The CRADA committed SOC-400 was under construction and was shipped to LMES the following week. The directional reflectometers and imaging spectroradiometers were also evaluated and the MIDAC facilities at Irvine, CA were visited. The return trip was by way of CCAD, Corpus Christi, TX to deliver the upgraded SOC-400 to the U. S. Army, familiarize them to its operation, and evaluate their applications. CCAD is the primary helicopter repair facility in the U. S. and their application was similar to that of NASA in that they need to determine the effectiveness of their new aqueous cleaning facilities and monitor such things as rinse water contamination. Their substrates and contaminants are much more diverse than the interest of NASA and their appear to be capable of the challenge. This collaboration resulted in an invitation to give a paper and exhibit the SOC-400 at the Tri-Services Environmental Conference (Hershey, PA, May, 1996). A paper was published in the proceedings of this conference describing the performance of the SOC-400 and its application to cleaning problems at CCAD (Attachment 5).

The use of the MIDAC spectrometer was particularly fortuitous since it is a very rugged, compact, and economical instrument. MIDAC is located in Irvine, CA which is convenient to SOC for same day service, if necessary. The operating system (MIDAC/GRAMS/32) is a very up-to-date 32-bit Windows'95 system that is both versatile and exactly what a chemist expects to see in FTIRS software. The learning curve for a typical chemist is measured in minutes. On several occasions, the local MIDAC representative visited the LMES facilities to evaluate the SOC-400 relative to some potential application. In those cases he brought his own laptop computer operating system that guaranteed confidentiality between MIDAC and LMES and SOC. The CRADA SOC-400 was returned to SOC (June, 1996) evaluations and to demonstrate its capability at detecting trace explosives on surfaces. ANADIS Instruments contracted with SOC (September 1996) to represent the SOC-400 in The Netherlands. The LMES PI and the CRADA SOC-400 traveled to Utrecht, The Netherlands, to participate in HET INSTRUMENTS '96, a biannual exposition of laboratory and process control equipment, to assist and train ANADIS in demonstrating the SOC-400 at their exhibit (Attachment 6). The ANADIS exhibit also had the MIDAC sales representatives from the United Kingdom and from Germany, as well as the MIDAC European sales supervisor. These representatives are independent contractors to MIDAC that represent an international network of sales, hardware, and software support that is willing and able to Market this product..

A collaboration was established between LMES, SOC, and NASA/MSFC to compare the performance of the CRADA SOC-400 with the NASA SOC-400 and with an FTIR microscope with respect to addressing the calibration problem of detecting oil on sandblasted D6AC steel rocket motor casing material. NASA has extensive experience with quantitatively applying oil contamination to sandblasted metals and have a requirement that they distinguish Number 2 grease from CRC Silicone grease on this steel surface at the 30 mg m^{-2} level. The experiments at NASA/MSFC compared the NASA SOC-400 to a Nicolet FTIR microscope and to other NASA techniques for contamination detection. The results of these experiments were written up and submitted to *Spectroscopy* for publication by SOC. A draft of this paper (Attachment 7) is attached. This paper is presently being revised after review, a process that has been delayed by the untimely death of Dr. Neu (October, 1996). NASA was very please with the results and claimed that they had detected contaminants that were known to be present, but that had never been detected by their other existing methods of analysis. NASA contamination standards (called step-plates) for Number 2 grease and for CRC Silicone grease were provided to LMES for spectral mapping analysis. The results demonstrated that the two SOC-400's performed comparably and were more than adequate to meet the intent of the original specification. These results were presented at the NASA Aerospace Environmental Conference (Huntsville, AL, August, 1996) and are being published as a paper in the proceedings of that meeting (Attachment 8). SOC also presented a paper on image analysis using scanning imaging spectroradiometers with applications to surface analysis.

The CRADA SOC-400 was taken to Lockheed Martin Aeronautics (Marietta, GA, May, 1996) to demonstrate its performance for the purpose of comparison to the SOC-100 directional reflectometer that is used to certify the paint finish on witness coupons for the F-22 advanced fighter aircraft. Their interest was to find a device that would make directional reflectance measurements on the aircraft. The SOC-400 is an approximation for a directional reflectometer with the angle fixed near zero degrees. Classification issues and a short time to production made the application of the SOC-400 to this problem difficult. However, the interest in an *in situ* paint certification was strong and the development a portable infrared reflectometer to compare with the SOC-100 coupon certification during the F-22 production may have strong long term possibilities. The aerospace industry was also addressed by a paper presented at the SAMPE Technical Conference (Seattle, WA, November, 1996), and published in the proceedings (Attachment 9). This paper described spectrometric imaging analysis for the determination of surface cleanliness and the detection of stains. Considerable interest was shown in this application to cleanliness certification and to the determination of heat damage on graphite-epoxy laminates.

The AMTEX CRADA for Finish on Yarn (FOY) evaluated the SIMIR for its ability to analyze cloth, individual threads, and chemical contaminants (finishes) on those materials and found considerable promise. Upon receiving the CRADA SOC-400 (April, 1996), it was immediately taken to the AMOCO Fabrics and Fibers Company Research and Development Center (Atlanta, GA) for a successful demonstration of its capabilities at a meeting of the AMTEX CRADA FOY investigators. The following month it was taken to the Hoescht-Celanese Dreyfus Laboratories (Charlotte, NC) at the request of AMTEX CRADA members for specific evaluations. The CRADA SOC-400 was subsequently taken to Cookson (Bristol, TN) and AMOCO (Atlanta, GA) for week long evaluations by individual experimenters. The SOC-400 performed certain tasks very well and represents a significant improvement over existing technologies. These AMTEX CRADA FOY exercises were funded through the AMTEX CRADA and the results are reported in the AMTEX CRADA FOY completion report.

A comparison of spectral mapping using the SOC-400 to the data obtained by scanning imaging spectroradiometers indicated that a sensitive imaging spectroradiometer with the sensitivity of the SOC-400 would be a very powerful tool for rapidly analyzing large quantities of bulk materials such as mixtures of recycle chips of plastics, paper, and wood. At present, such a imaging spectroradiometer would be extremely expensive and at the edge of the present state-of-the-art. However, SOC-400 spectral mapping can produce images which have the required spectrometric quality to evaluate the possibilities of chemical discrimination of mixtures and these images can be analyzed by several levels of chemometric techniques and by the techniques typically used for color discrimination on spectroradiometer images. An OIT Forest Products CRADA involving LMES, Sandia National Laboratories, and this CRADA SOC-400 was proposed, accepted, and mid-infrared spectral images of various paper and wood products are being successfully obtained.

The life of the CRADA paralleled the implementation of the DOE-DP Enhanced Surveillance Program, which strongly supported the development and application of this technology. SOC-400s have been acquired by LMES, LLNL, LASL, and Pantex for the inspection of surfaces for corrosion and residual organic compounds. Formal and informal collaborations have been established to explore applications, establish defined inspection criteria, develop automated mapping and data reduction techniques, and solve short-term problems. Progress has been made in modifying the SOC-400 for a higher capability for concave surfaces such as inside cups and pipes. These exercises were funded through DOE-DP programs and will be reported in detail through those channels.

INVENTIONS

There were no inventions made or reported as a result of this CRADA.

COMMERCIALIZATION POSSIBILITIES

The SOC-400 is now a commercial device in the international market place. Six SOC-400 units were sold in CY 1996. The annual market potential should be greater than that in the future, but depends primarily on the ability to tailor the SOC-400 to a particular application such that the customer sees the instrument as a turn-key device.

PLANS FOR FUTURE COLLABORATION

LMES and SOC participants have verbally agreed to continue the CRADA as a means of expanding the capabilities of the SOC-400, particularly with respect to developing inspection techniques and improving the performance of the SOC-400. LMES will continue to use the SOC-400 provided for this CRADA in support of the OIT/Forest Products CRADA.

CONCLUSIONS

This CRADA has been successful at establishing the SOC-400 Surface Inspection Machine-Infrared (SIMIR) as a viable product in the international market place and as an essential tool in DOE-DP Enhanced Surveillance activities. This CRADA also has provided that critical element of having an SOC-400 available to evaluate real capability for immediate decision making in the light of the needs of the DOE-DP Enhanced Surveillance Program, the textile industry, the forest products industry, the aerospace industry, and the private sector in general.

ATTACHMENTS

Attachment 1. G. L. Powell and T. E. Barber, "Inspection of Surfaces in an Assembly Dry Room," *Lockheed Martin Energy Systems Report Y/DZ-1114*, Oak Ridge, Tennessee August 3, 1996.

Attachment 2. Specification for the SOC-400 Surface Inspection Machine-Infrared (SIMIR). This specification differs from the original specification used by NASA/MSFC only by the use of current computer specifications and in the application of statistical limits on the noise requirements.

Attachment 3. Abstract from Pittcon'96, the premier conference and exposition on laboratory equipment and chemical analysis, Chicago, IL, March, 1996.

Attachment 4. Surface Optics Corporation sales brochure for the SOC-400 Surface Inspection Machine-Infrared (SIMIR), March, 1996.

Attachment 5. G. L. Powell, E. G. Engberg, J. Holiday, and J. L. Velez, "Application of Mid-Infrared Spectroscopy to the Identification of Materials and to the Determination of Surface Coatings," Tri-Services Environmental Workshop, D. Bader, ed., AEC, APG, Maryland, 25-34 (1996). cycled separately as Y/DZ--2009

Attachment 6. ANADIS Instruments sales brochure for the HET Instruments exhibition, Utrecht, the Netherlands, September, 1996.

Attachment 7. G. L. Powell, B. H. Nerren, R. Booth, and J. T. Neu, "Contamination Measurement in the Manufacture of Solid Rocket Casings using a Portable FTIR Spectrometer," Spectroscopy, in press. preprint removed.

Attachment 8. G. L. Powell, T. E. Barber, J. T. Neu, and B. H. Nerren" Diffuse Reflectance Mid-infrared Spectroscopy as a Tool for the Identification of Surface Contamination on Sandblasted Metals," Aerospace Environmental Technology Conference, NASA Conference Publication, A. F. Whitaker, ed., MSFC, Alabama, (1996), in press. cycled separately as Y/DZ--1174/R1

Attachment 9. G. L. Powell, R. M. Cox, T. E. Barber, and J. T. Neu, "Nondestructive Inspection of Organic Films on Sandblasted Metals using Diffuse Reflectance Infrared Spectroscopy," 28th International SAMPE Technical Conference 28, Covina, California, 1171-1182 (1996). cycled separately as Y/DZ--1193

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3. G. L. Powell, M. Milosevic, J. Lucania, and N. J. Harrick, "The Spectropus System, Remote Sampling Accessories for Reflectance, Emission, and Transmission Analysis using Fourier Transform Infrared Spectroscopy," *Appl. Spectrosc. 46*, 111-125 (1992).

4. G. L. Powell, N. R. Smyrl, C. J. Janke, E. A. Wachter, W. G., Fisher, J. Lucania, M. Milosevic, and G. Auth, "Nondestructive Inspection of Graphite-Epoxy Laminates for Heat Damage using DRIFT and LPF Spectroscopies," in *The Proceedings of the Conference on Characterization and NDE of Heat Damage in Graphite Epoxy Composites, NTIAC*, Austin, Texas, 97 - 111 (1993).

5. G. L. Powell, N. R. Smyrl, D. M. Williams, H. M. Meyers, III, T. E. Barber, M. Marerro-Rivera, "Surface Inspection Using Fourier Transform Spectroscopy," *Aerospace Environmental Technology Conference, NASA Conference Publication 3298*, A. F. Whitaker, ed., MFSC, Alabama, 563-571 (1995).

6. G. L. Powell, T. E. Barber, and J. T. Neu, "Instrumentation for the determination of Material Properties from Spectroscopic Measurements of Total Integrated Scatter," *Proceedings of SPIE's 40th Annual Meeting and International Symposium on Optical Science, Engineering, and Instrumentation 2541*, J. C. Stover, ed., Bellingham, Washington,142 - 153 (1995).

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Attachment 1. G. L. Powell and T. E. Barber, "Inspection of Surfaces in an Assembly Dry Room," *Lockheed Martin Energy Systems Report Y/DZ-1114/R1*, Oak Ridge, Tennessee September 9, 1996.

Y/DZ-1114
Abstract

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INSPECTION OF SURFACES IN AN ASSEMBLY DRY ROOM

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INSPECTION OF SURFACES IN AN ASSEMBLY DRY ROOM

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The INSPECTOR™, a portable Fourier transform infrared spectrometer for surface analysis by diffuse reflectance, was placed in an assembly dry room to demonstrate its use in a production environment. The primary object was to analyse LiH for LiOH as a measure of prior moisture exposure. The spectrometer was operated in a manner analogous to a top-loadmg balance that obtained infrared spectra from whatever was placed on its face-plate. A number of parts and coupons were analyzed along with other materials such as paper. LiOH formation over time on previously baked LiH as a result of exposure to the typically 5-ppm water vapor in the environmentally controlled room is shown in Figure 1. The exposure consisted of leaving the specimen on the spectrometer sample stage while the spectrometer was not purged. Spectra were taken hourly for three days. The spectra give the reflectance in Kubelka-Munk units. These units may be scaled by the factor of 3900 mg m^{-1} per Kubelka-Munk unit from ref. 1. This indicates a detection limit of 1 mg m^{-2} and a reaction rate of ~2 mg m^{-2} hr^{-1}. The band at 3675 cm^{-1} is that for normal LiOH that typically forms on the surface of as-machined LiH. The band at 3528 cm^{-1}

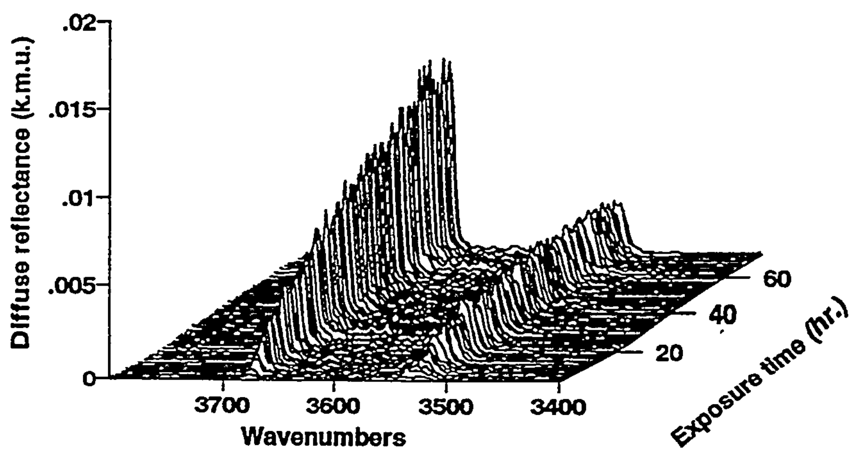

Figure 1. LiOH formation over time on previously baked LiH as a result of exposure to the typically 5-ppm water vapor in the environmentally controlled room.

forms only on LiH that has been vacuum bake and occurs at a lower frequency than that of $LiOH \cdot H_2O$ (3566 cm^{-1}). The particular structure of this hydroxide and its limited ultimate growth relative to normal LiOH (3675 cm^{-1}) is probably due to reaction with defect sites in the initial coating of Li_2O that was formed from the decomposed a surface film of LiOH during the vacuum bake. This existence of this specific form of LiOH was not known prior to these experiments.

[1] G. L. Powell, M. Milosevic, J. Lucania, and N. J. Harrick, "The Spectropus System, Remote Sampling Accessories for Reflectance, Emission, and Transmission Analysis using Fourier Transform Infrared Spectroscopy," *Appl. Spectrosc.* **46**, 111 - 125 (1992).

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Attachment 2. Specification for the SOC-400 Surface Inspection Machine-Infrared (SIMIR). This specification differs from the original specification used by NASA/MSFC only by the use of current computer specifications and in the application of statistical limits on the noise requirements.

SIMIR SPECIFICATION

Herein is a description of a surface inspection machine (SIMIR) based on a portable Fourier transform infrared spectrometer that uses diffuse reflectance optics to perform surface inspection operations in manufacturing environments, including inspection for surface cleanliness, dryness, oxidation, or contamination; materials identification; and characterization of adhesives, polymers, and composite materials. This SIMIR consists of the integration of a diffuse reflectance optical sampling device with a small Fourier transform infrared spectrometer, including computer and operating system in such a way as to maximize the performance, minimize the size and weight, and yield a robust instrument capable of use in a manufacturing environment. The SIMIR shall be a Surface Optics Corporation (San Diego, CA) SOC-400 or approved equal.

The SIMIR optical probe shall be as small and light as possible, no larger than 11-in. by 10-in. by 9-in. and weigh no more than 18 lbs. One side of the SIMIR shall contain the sample position of a diffuse reflectance optical sampling device and shall analyze concave surfaces with radii of curvature greater than 0.25 m, flat surfaces, and convex surfaces, regardless of their other dimensions or the orientation of the sample in space. The SIMIR shall be robust with respect to motion and vibration and shall be operable up to accelerations of 0.1 g due to vibrations within the entire acoustic spectrum. The SIMIR shall be connected to electrical power, computer, and purge gas by a set of flexible cables including power cable, computer communication line, and purge gas line so that the inspection machine can be operated up to 100-ft. from the supporting utilities (computer, purge gas supply, and 120 VAC-60 Hz electrical power). The instrument shall also be operable directly from a 12 VDC automotive type battery at 10-ft. The SIMIR shall require no utilities other than those mentioned in the previous sentence, shall require no more than 50 watts of electrical power, and shall require no internal mechanical modifications prior to shipping. The inspection machine shall have a sampling face plate of metal or plastic that is readily replaceable with modifications to adapt the SIMIR to a particular inspection task. The SIMIR shall have the capability for attaching a remote detector and an external optical head, with each item to be purchased separately.

The spectrometer shall provide a collimated infrared beam that has been modulated by a Michaelson interferometer capable of 2, 4, 8, 16, and 32 wavenumber resolutions. The "hot" source (source temperature > 1500 K) will be used. The collection optics shall be configured for diffuse reflection that is 99.5% effective at rejecting nominally specularly reflected light, and that shall collect light over 65% of the solid angle that subtends scattered light and deposit this collected light on a DTGS detector. Ninety percent of the incident light should be focused on a spot <2 mm. in diameter on the sampling surface.

The SIMIR operating as a unit with the contact face plate against a planar, sand-blasted aluminum surface shall have a sensitivity of 0.0001 absorbance units defined as the 95% confidence limit of a smooth curve fitted through 500 wavenumber segments of the spectrum within the 600 wavenumber-to-3000 wavenumber range when the spectrum is collected over a time interval of 1 minute at 16 wavenumber resolution and 2X zero filling following a background spectrum obtained from the same surface.

Attachment 3. Abstract from Pittcon'96, the premier conference and exposition on laboratory equipment and chemical analysis, Chicago, IL, March, 1996.

Y/DZ-1170
Abstract

Y-12

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LOCKHEED MARTIN

APPLICATION OF MID-INFRARED SPECTROSCOPY TO THE IDENTIFICATION OF MATERIALS AND TO THE DETERMINATION OF SURFACE COATINGS

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APPLICATION OF MID-INFRARED SPECTROSCOPY TO THE IDENTIFICATION OF MATERIALS AND TO THE DETERMINATION OF SURFACE COATINGS

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Mid-infrared spectroscopy is a very useful tool for the determination of surface contamination in manufacturing applications. Due to the elimination of halogenated cleaning solvents, new cleaning processes are being implemented. Because of the uncertainty of the effectiveness of new processes, it is often necessary to verify the cleaning of a surface prior to continuing the process. This presentation describes the performance of a hand-held (or remotely positioned) Fourier transform infrared spectrometer that uses diffuse reflectance optics to interrogate surfaces. The Surface Optics Corporation SOC 400 Surface Inspection Machine / InfraRed (SIM/IR)[1] shown in Figure 1 weighs less than 8 Kg and may be manipulated into any orientation during operation. The SIM/IR is based on barrel ellipse diffuse reflectance optics having a focal point located in the plane of an opening in the face plate of the instrument. The system is supported by either 12 VDC or 120 VAC and a computer using MIDAC GRAMS/386. This "point-and-shoot" type of instrument allows rapid in-field analyses to be made with sensitivity comparable to sample compartment accessories, dedicated scanning and collection routines, and the robust characteristics of the barrel ellipse focal point with respect to sample positioning. The capability for having the spectrometer software instruct positioning devices in a noncontact mode is utilized. Applications to inspecting sandblasted metal surfaces for oil, automobile finishes, paper, thread, and graphite-epoxy laminates, will be described.

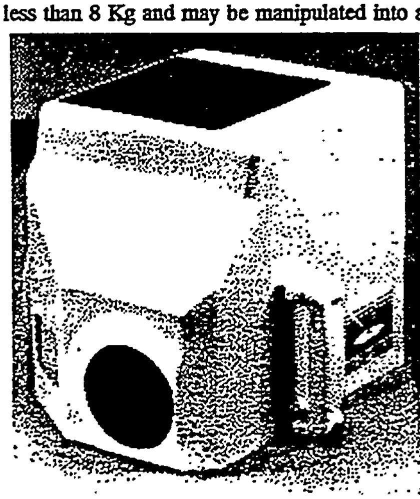


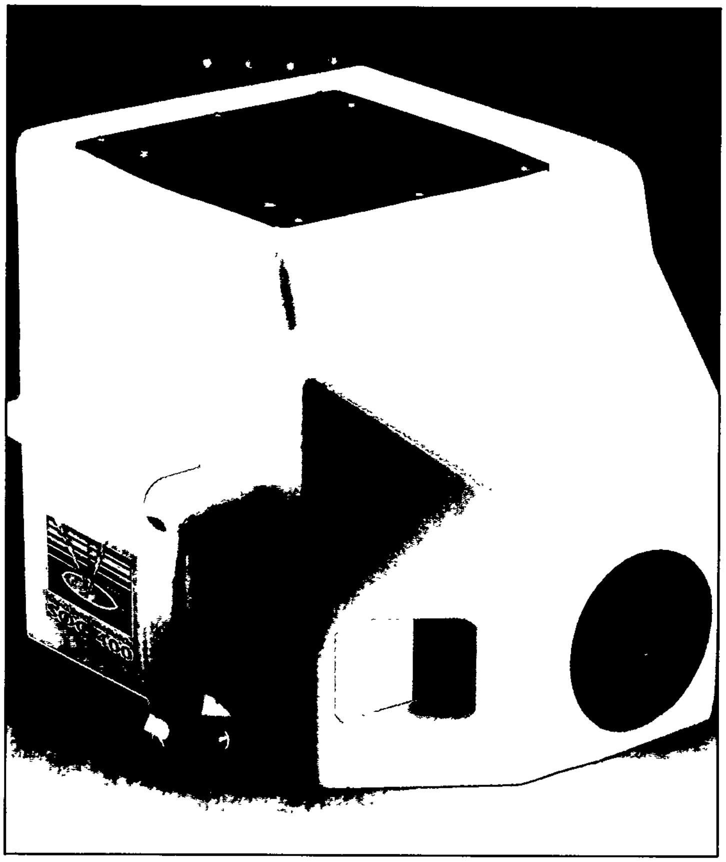
Figure 1. The SOC 400 Surface Inspection Machine/InfraRed (SIM/IR). The sampling point of the diffuse reflectance optics is at the center of 85-mm diameter dark circle.

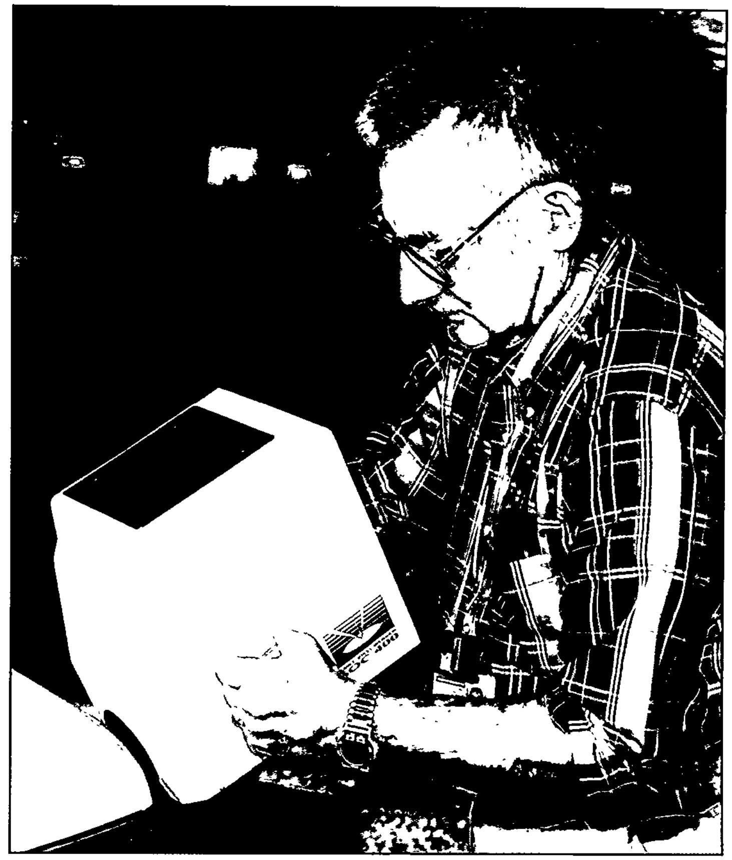
[1]G. L. Powell, T. E. Barber, and J. T. Neu, "Instrumentation for the Determination of Material Properties from Spectroscopic Measurements of Total Integrated Scatter," *Proceedings of SPIE's 40th Annual Meeting and International Symposium on Optical Science, Engineering, and Instrumentation 2541*, J. C. Stover, ed., Bellingham, Washington, 142 - 153 (1995).

The authors gratefully acknowledge B. H. Nerren, National Aeronautics and Space Administration, Marshall Space Flight Center, Alabama, United States for his efforts to implement this inspection technique.

[*]Managed for the U. S. Department of Energy by Lockheed Martin Energy Systems, Inc. Under Contract No. DE-AC05-84OR21400.

Attachment 4. Surface Optics Corporation sales brochure for the SOC-400 Surface Inspection Machine-Infrared (SIMIR), March, 1996.





SOC-400 SURFACE MEASUREMENT SYSTEM

"THE SOC-400 IS A LONG NEEDED TOOL FOR QUALITATIVELY AND QUANTITATIVELY DETERMINING SURFACE CLEANLINESS, DEGRADATION, AND OTHER SURFACE AND NEAR SURFACE PROPERTIES."

Bill Nerren, NASA/MSFC

Quality diffuse FTIR spectrometer scattering measurements, which heretofore were only available on samples in the laboratory, may now be measured in-situ at the sample site in the field or factory, etc. The SOC-400 is a small handheld diffuse reflectometer. It has a signal-to-noise greater than 5000 and can potentially identify literally millions of chemical species on surfaces. Its development will allow the application of state-of-the-art infrared spectroscopy to real surfaces in practical environments as a nondestructive inspection process.

Using the SOC-400 surface measurement system, state-of-the-art FTIR diffuse scattering spectra may be obtained on material surfaces for which it is impractical or impossible to obtain specimens for laboratory study. The measurement is simply accomplished by placing the integral FTIR-optics measurement head in contact with the sample surface and directing the measurement operation from a computer in a fashion exactly analogous to a laboratory FTIR measurement. The measurement head is designed to be held in place for measurement operation by hand, or by a special positioning fixture. The measurement head is prealigned for surface mounted operation for any physical orientation of the instrument. The spectroscopic performance of the total system is optimized for maximum signal-to-noise and is comparable to laboratory devices.

The SOC-400 measures diffuse reflectance spectra of surfaces relative to an idealized surface. The instrument was developed under contract with NASA Marshall Space Flight Center to meet a critical requirement for measurement of contaminants on the inside surfaces of solid rocket motor castings. The list of types of materials which can be identified qualitatively or quantitatively is extensive, including powders, translucent organic solids (wood, paper, coal, etc.), graphite-resin composites, organic and silicone oil films on metals.

The SOC-400 has the unique capability to measure these materials in-situ on flat or curved surfaces and makes practical the process of monitoring optical changes in surfaces over a period of time when exposed to various elements. This capability is particularly useful for evaluating vehicles and aircraft coatings for quality and environmental degradation by comparison of measurements taken over a period of time.

SURFACE OPTICS CORPORATION

TEL: (619) 675-7404

11555 Rancho Bernardo Road, San Diego, CA 92127-1441

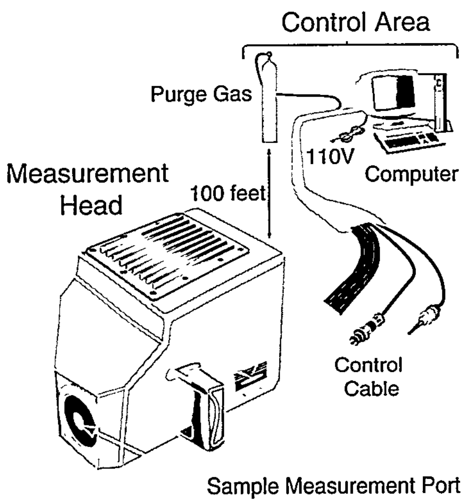
http://www.surfaceoptics.com

E-mail: soc@surfaceoptics.com

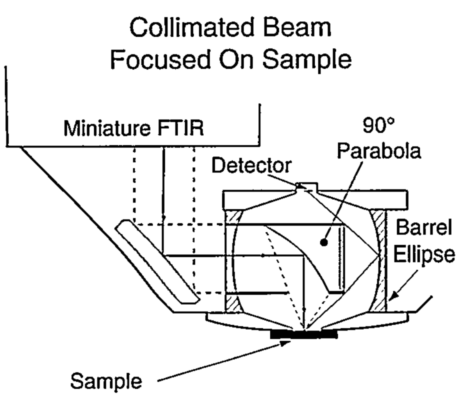
FAX: (619) 675-2028

SOC-400 SYSTEM

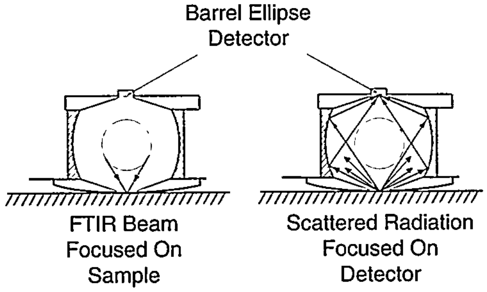
The SOC-400 is a portable system built to function in diverse environments to measure diffuse reflectance on a wide variety of surface types in field locations. It is composed of the measurement head connected by a control cable to a computer platform and purge gas system. It may be mounted in any required position to measure the sample surface of interest.



Source Optics: The head consists of a barrel ellipse which collects scattered radiation. The collimated beam from the interferometer enters this ellipse along its minor axis and is focused by a parabolic mirror, supported through the opening, to a 2 mm diameter spot centered at one focus of the barrel ellipse in the center of the measurement port.



Detector Optics: By placing the measurement head port against the sample surface, the barrel ellipse images onto the detector the scattered radiation from the sample. Specularly reflected radiation is blocked by the parabolic mirror and not recorded. The precision barrel ellipse optic is small, minor axis 2½", foci separation 2", to facilitate placing the measurement head in confined spaces. The detector is an advanced DLATGS thermal sensor which does not require cooling.



SPECIFICATIONS

MEASUREMENT HEAD	
Wavelength Coverage	5000-400 cm^{-1} 2.0-25 μm
Resolution	2,4,8,16,32,cm^{-1}
Weight	18 lbs.
Dimensions	9.75" H, 8.25" W, 11" D
Sensitivity	~0.0001 absorbance units*
CONTROL STATION	
Computer	IBM compatible, desktop or laptop
Software	MIDAC GRAMS 386
Purge Source	0.2 cfm Dry Co_2 Free Gas Cylinder

*Sensitivity is based on the ability to detect the 2925 cm^{-1} hydrocarbon band on sandblasted gold in one minute (48 scans) collection time at 16 cm^{-1} and 2X zero-filling.

APPLICATIONS

Three representative examples of the large number of possible applications are noted below:

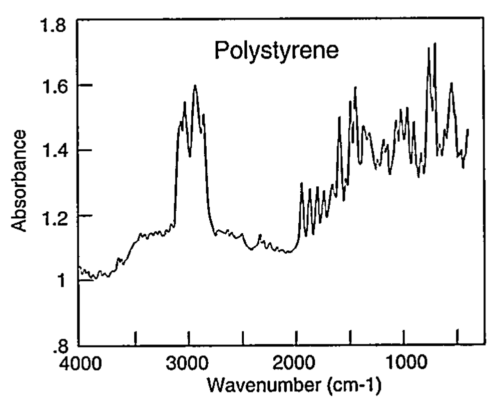
Surface Inspection: The SOC-400 was initially developed for surface inspection applications. It provides an outstanding capability to detect, identify and quantify surface contaminants. Films in the nanometer range may be detected. Chemical composition of films in the thickness range from 100 nm to 100 micrometers may be determined.

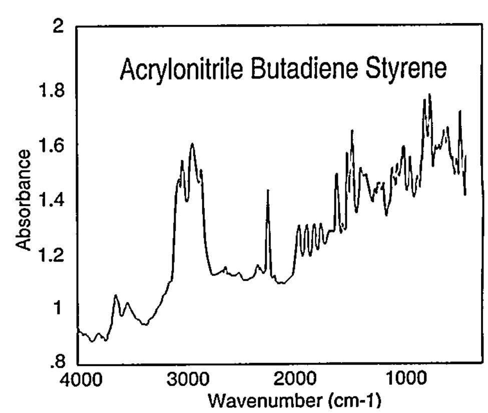
Matrix Mapping of Surfaces: Flat or curved sample surfaces may be mapped using automated hardware coupled with the SOC-400 to scan the sample in a programmed pattern. Large numbers of spectra can be collected over time from a known grid and catalogued in a single data file or data base. Tables of data, such as baseline corrected peak heights, can be produced from thousands of spectra in the data base. The MIDAC/GRAMS 386 operating system and supporting software can display these spectra as 3-D graphs, contour maps, movies, etc.

As an example, by identifying spectral changes in graphite epoxy laminates, areas of heat induced strength degradation may be mapped by a nondestructive method.

Process Control: The SOC-400 is also suitable for process control applications. As an example, for plastics in granular form there are requirements to identify trace granules of one chemical structure mixed with a small quantity of granules of different chemical structure. Since the measured spot size of the SOC-400 is smaller than the nominal size of the granules, the reflectometer may be used to distinguish between two (or more) types of plastics.

In the figures below, the spectra of two plastics of similar chemical structure, polystyrene and acrylonitrile butadiene styrene measured with the SOC-400 are shown below. In this representative example of commonly used plastics, the traces are seen to be similar except the strong C≡N band of the acrylonitrile butadiene styrene sample is absent in the polystyrene sample, providing unambiguous identification.





Attachment 5. G. L. Powell, E. G. Engberg, J. Holiday, and J. L. Velez, "Application of Mid-Infrared Spectroscopy to the Identification of Materials and to the Determination of Surface Coatings," Tri-Services Environmental Workshop, D. Bader, ed., AEC, APG, Maryland, 25-34 (1996).

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Attachment 6. ANADIS Instruments sales brochure for the HET Instruments exhibition, Utrecht, the Netherlands, September, 1996.

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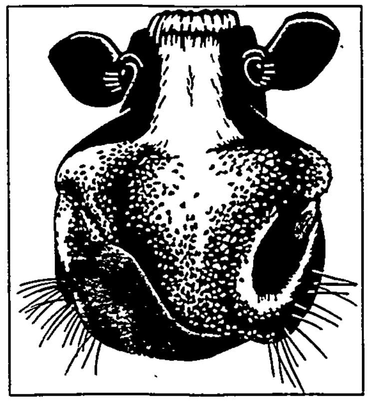

INTRO:

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SOC-400 Surface measurement system

"The SOC-400 is a long needed tool for qualitatively and quantitatively determining surface cleanliness, degradation, and other surface and near surface properties." Bill Nerren, NASA/MSFC

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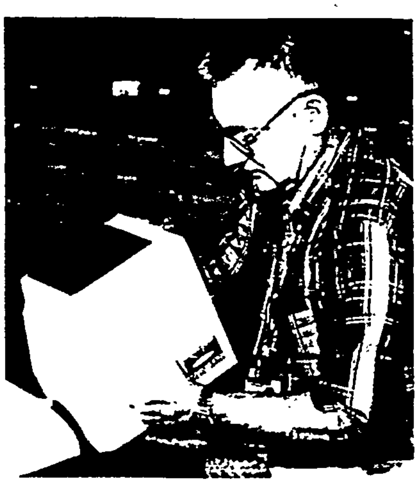

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precision and accuracy necessary for true comparison of spectral data and/or for development of quantitative methods.

Quant Print automatically applies the sample evenly over the defined target area of the card, therby providing a reproducible pathlength. The system offers three deposition speeds for samples of different viscosity.

The PIKE Quant-Print Sample Deposition System is extremely easy to use. The procedure simply involves the placement of a 14 microliter sample drop to the center of the applicator piston, and insertion of a card. All the remaining steps are controlled automatically by an optical switch and system electronics. Quant-Print in combination with 3M IRCards is well suited for analysis of engine oils, automotive fluids, mineral oils, edible oils and other hydrophobic samples.

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Starna levert al sinds het begin van de jaren vijftig cuvetten van prima kwaliteit. De gunstige prijs geeft naast vakkundig advies prijskwaliteitverhouding als meerwaarde. Cuvetten: stan daard vierkante met platte deksel, stop of schroefdop - cylindrische - doorstroom - korte en lange weglengte - ultramicro, micro, semimicro of macro, enz. Van het uitgebreide leverings programma is een populaire selektie beschreven in de recent uitgegeven 32 pagina tellende brochure. M.b.v. bijgaande antwoordkaart kunt u deze brochure aanvragen, waarna wij voor prompte toezending, vergezeld van prijslijst, zullen zorgdragen.

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Easy sampling:

- Turn knob down untill required pressure (read on scale);
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Featuring:

- Two silicon hemispheres in removable sample holders either in sealed top-plate (excellent S/N ratio of 20 micron and big samples as well, sampling/cleaning in- or outside the instu ment);
- Silicon advantage (Hard, cheap, IR transparent also because of reflective transfer optics 1000-100cm^{-1}, high n, etc.);
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- High throughput (for best S/N) macro, 600 micron sampling area, sampling elements
- Vieuwing or non vieuwing pressure tool;
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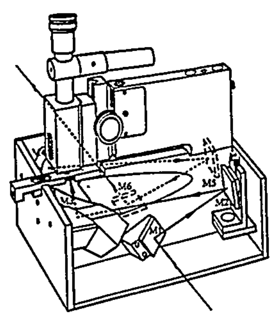
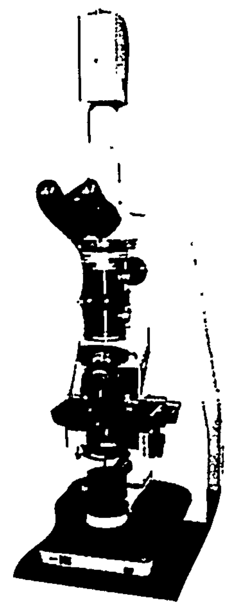


Chalenging FTIR Microscopy



3 New IR microscopes from Aire Scientific. Illustrated above is the stand alone 'Discovery'.

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Attachment 7. G. L. Powell, B. H. Nerren, R. Booth, and J. T. Neu, "Contamination Measurement in the Manufacture of Solid Rocket Casings using a Portable FTIR Spectrometer," Spectroscopy, in press.

preprint removed.

Attachment 8. G. L. Powell, T. E. Barber, J. T. Neu, and B. H. Nerren" Diffuse Reflectance Mid-infrared Spectroscopy as a Tool for the Identification of Surface Contamination on Sandblasted Metals," Aerospace Environmental Technology Conference, NASA Conference Publication, A. F. Whitaker, ed., MSFC, Alabama, (1996), in press.

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Attachment 9. G. L. Powell, R. M. Cox, T. E. Barber, and J. T. Neu, "Nondestructive Inspection of Organic Films on Sandblasted Metals using Diffuse Reflectance Infrared Spectroscopy," 28th International SAMPE Technical Conference 28, Covina, California, 1171-1182 (1996).

Y/DZ--1193 cycled separately.