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MCC Case Study

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MISSION:

The *Materials Characterization Center, Inc. (MCC)* strengthens the relationship between industry and academia and thereby enhances the environment for scientific research and development in Puerto Rico, by providing state-of-the art analytical services and scientific expertise, not readily available through commercial laboratories, to clients in industry, academia and government.

VISION:

The *Materials Characterization Center, Inc. (MCC)* envisions itself a non-profit, self-supporting entity which strengthens and improves Puerto Rico's scientific and technological infrastructure. This infrastructure is important in retaining and attracting new high technology industries and thus contributing to the economic development of Puerto Rico.

CHARACTERISTICS AND BENEFITS

- ❑ Techniques include:

  Nuclear Magnetic Resonance (500 and 300 MHz)
  Mass Spectrometry (HPLC-MS, GC-MS, FAB, Others)
  Surface Microscopy and Spectroscopy (SEM-EDS, FT-IR, XPS, AFM. Others)
  X-Ray Diffraction and Crystallography (powders Polarized microscopy, DSC, TGA)

- ❑ Scientific and Technical expertise from experts in each field
- ❑ Interdisciplinary collaboration among laboratories
- ❑ Competitive costs, expedient service
- ❑ Strict confidentiality
- ❑ FDA Registration / GMP compliance
- ❑ Training available for your staff
- ❑ Solve problems in manufacturing processes, in quality control, in facilities, packaging materials, and many other applications.

SOME COMMON USES AND APPLICATIONS

- Determination of the chemical structure of an unknown compound
- Analytical determination and characterization of impurities in mixtures
- Chemical and physical characterization of pharmaceutical solids
- Elemental and molecular composition of surfaces
- Identification of contaminants in solids
- Identification of polymorphs in pharmaceuticals solids
- Determination of solvent and raw material purity
- Characterization of unexpected HPLC peaks

We focus on quality and speedy turn around times tailored to demanding industrial deadlines.

- Characterization of gases and odors
- Headspace analysis
- Trace analysis

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CASE STUDIES OF SUCCESFUL PROJECTS

Note: All projects performed by the Materials Characterization Center are strictly confidential in nature. In the descriptions of the following case studies, names of firms and origin of samples are not mentioned. However, from the information furnished, one may be able to speculate about the sample's source. These case studies are thus deemed confidential and prepared for the sole purpose of informing a selected audience of the Center's activities.

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Case Study I

Problem: A user of computer boards identified two seemingly unrelated problems within days of each other: some computer boards were exhibiting faint marking or spots on the surface which were unacceptable to clients and secondly, sometimes boards did not respond when inserted in the box but on removing and reinserting they would function properly.

Experiment: Scanning Electron Microscopy (SEM) of the board's surface revealed the extent of the markings. Simultaneous Energy Disperse X- Ray Spectrometry (EDS) of these showed chlorine and Bromine residues over the surface and in the gold plated connectors. Fourier Transform- Infrared Spectroscopy (FT-IR) confirmed the findings.

Solution: Chlorine and Bromine were traced back to the cleaning agent being used. The suggestion was to properly rinse boards after the washing cycle. Faulty connections were also caused by the presence of cleaning agent residues. After inserting and reinserting the connectors were mechanically "cleaned" and functioned properly.

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Case Study II

Problem: Tablets from a pharmaceutical operation showed slightly dark streaks during inspection in the production line.

Experiment: Observation under the Scanning Electron Microscope (SEM) revealed that the problem was merely superficial. By careful manipulation, part of the discolored surface was obtained and analyzed by High-Resolution Mass Spectrometry (MS) using the direct insertion probe (DIP) technique. The fragmentation pattern obtained suggested the presence of a hydrocarbon mixture and thus an oil was suspected as the culprit.

Solution: After obtaining samples of lubricating oils used at the plant, each was subjected to Mass Spectra analysis and the fragmentation patterns compared to that from the tablets. The particular oil on the tablet surface was thus identified.

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Case Study III

Problem: A bronze Astrolabe found alongside an underwater wreck of a Spanish galleon was being investigated for historical and judicial purposes. It was necessary to ascertain that the piece was genuine and that it dated back to the XVII Century.

Experiment: The surface of the Astrolabe was analyzed by Energy Disperse X-Ray Spectroscopy (EDS) and a elemental composition in terms of percent cooper, chromium, nickel and other metals was obtained.

Solution: Comparison of the metal composition revealed that the piece was indeed manufactured in Portugal during the XVII century since the type of bronze used by Portuguese craftsmen at that time is well documented.

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Case Study IV

Problem: Tablets manufactured by a pharmaceutical exhibited random black spots on the surface. The nature and origin of the black spots needed to be identified for regulatory compliance purposes.

Experiment: Under a conventional microscope, some black spots were carefully removed and analyzed by Fourier Transform Infrared Spectroscopy (FT-IR). A search with the computer library of the instrument suggested that the spots consisted of a polymer normally referred to as phenoxy resin. This polymer is used to manufacture black hard plastics.

Solution: With this information a search in the production lines for possible sources of phenoxy resin was initiated and several possibilities identified. The presence of phenoxy resin in the tablets was confirmed by an experiment showing that the substance is fluorescent as expected.