Designation: E 416 – 98 (Reapproved 2004)

Standard Practice for Planning and Safe Operation of a Spectrochemical Laboratory[1]

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1. Scope

1.1 This practice covers the general planning of a spectrochemical laboratory, the equipment necessary for efficient operation of such a laboratory, and recommended safety precautions to be considered. Principal equipment housed in such a laboratory may include optical emission spectrographs, vacuum and air-path optical emission spectrometers, plasma emission spectrometers, X-ray emission spectrometers, X-ray diffractometers, and atomic absorption and flame emission spectrophotometers.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

E 50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E 115 Practice for Photographic Processing in Optical Emission Spectrographic Analysis[3]

E 406 Practice for Using Controlled Atmospheres in Spectrochemical Analysis

E 528 Practice for Grounding Basic Optical Emission Spectrochemical Equipment[3]

3. Significance and Use

3.1 While considerations for spectrochemical equipment are a prime concern to spectroscopists, it is quite likely other analytical equipment and other chemical and physical laboratories will be in contiguous space. Therefore, the proximity of these other laboratories should be a primary concern in the location and design of a spectrochemical laboratory.

3.2 The safety and health of all individuals working in any laboratory facility is of primary importance. Therefore, the design of the laboratory and the equipment used therein should include the latest recommendations of safety and health experts. In addition, all laboratory facilities should meet all of the safety and health standards of federal, state, and local authorities.

3.3 This practice will be useful for plant managers, architects, design or construction engineers, or anyone involved with the design, construction, or remodeling of laboratory facilities. Purchasing agents, chief spectroscopists, or anyone responsible for the purchase and location of new equipment will also find this practice useful.

4. Location

4.1 The general location of the laboratory depends primarily on whether it is to serve as a research, commercial, government, or a production facility. The laboratory should preferably be near the organization it serves or near the principal source of samples. If this is not possible, consideration should be given to a means of rapid transportation of samples from the source to the laboratory. Vibration, dust, fumes, noise, humidity, and temperature fluctuations should be at a minimum to ensure maximum capabilities, precision, and employee health. These factors will be considered later.

5. Arrangement

5.1 *Space Allocation*—The arrangement of the rooms for spectrochemical analysis shall provide easy access, semiprivacy, and secondary exits. Space for additions and modifications to equipment should be considered. The chief spectroscopist should be consulted on any laboratory planning, whether a large area or a small addition is under consideration.

5.2 *Optical Emission Spectrographic and Spectrometric Equipment*—The equipment will consist of various combinations of excitation sources, spectrographs or spectrometers, excitation stands, and in some cases, gas-handling apparatus and special sample holders that are described in the various spectrochemical methods. If possible, this equipment should be

[1] This practice is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores and Related Materials and is the direct responsibility of Subcommittee E01.20 on Fundamental Practices.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Withdrawn.

placed in an instrument laboratory away from the more corrosive atmosphere of a general chemical laboratory to avoid salt coating of optics and the corrosion of electrical components.

5.3 *Photographic Processing Equipment*—Photographic instruments will require a darkroom and equipment as described in Practice E 115. A refrigerator or freezer is desirable for film or plate storage. The spectrochemical darkroom shall not be shared with other facilities.

5.4 *Measuring and Calculating Equipment*—A microphotometer will be required in those installations using films or plates. This equipment is best housed in a separate room where the lights can be dimmed. Film viewers, calculating boards, desk calculators, microprocessors, and computer terminals will be required, depending upon the size and scope of the installation.

5.5 *Plasma Spectrometers*—Inductively coupled, d-c, and microwave-induced plasma spectrometers should be placed in an instrument laboratory separate from the general chemical laboratory for the same reasons outlined in 5.2.

5.6 *X-Ray and Optical Emission Instruments*—Place this equipment near the general chemical laboratory, but not in the same room. Avoid placing X-ray equipment in the same room with optical emission and inductively coupled plasma equipment in order to prevent radio-frequency electrical disturbances to the X-ray equipment. Federal, state, and local regulations with regard to shielding, safety interlock switches, and other devices for the control of radiation should be observed when laboratory space is designed for this equipment.

5.6.1 Instruments sensitive to electric line surges or line noise should be isolated from electrical lines serving furnaces, motors, or other surge-producing equipment. Electrical line filters or autotransformers may help reduce or eliminate electrical noise.

5.7 *Atomic Absorption and Flame Emission Spectrophotometric Equipment*—Atomic absorption spectrophotometers and flame photometers should be placed in a separate area adjacent to the general chemical laboratory. However, if space is limited, they could be placed in the general chemical laboratory.

5.8 *Sample Preparation*—A separate room for sample preparation should be provided with adequate space for such items as a bench grinder, lathe, drill press, cut-off machine, milling machine, button-melting furnace, electric mortar and pestle, screening and mixing equipment, briquetting press, electrode shaper, band saw, belt sander, and various hand tools. A low-power binocular microscope should be available to facilitate the removal of foreign material from matrix materials. The sample preparation room and each piece of dust-producing equipment should be provided with adequate dust removal to prevent the accumulation of potentially harmful dust and noxious fumes.

5.9 *Chemical Facilities*—The spectroscopist may require many of the facilities of the chemical laboratory; therefore, ideally, he/she should have available a chemical bench, sink, hot plate, oven, muffle furnace, fume hood, analytical balance, chemical glassware, etc.

5.10 *Workbench*—The spectroscopist will ordinarily require storage cabinets for chemicals, reference materials, and samples at a workbench provided with a dental amalgamator for mixing specimens and reference materials. This workbench should be near the spectrochemical equipment.

5.11 *Storage*—Space should be provided for storing reference materials, samples that must be retained, reference films and plates, strip charts, record books, report sheets, computer data, etc.

5.12 *Communication*—Telephone, teletype, pneumatic tube, a computer data-handling system, or similar systems shall be provided for transmitting samples or results, as required.

6. Construction

6.1 *Walls, Floors, and Work Surfaces*—Walls, floors, and work surfaces shall be finished with materials that are easily cleaned and maintained. Paints used on walls and laboratory furniture as well as materials used for work surfaces and floor coverings shall be resistant to the chemicals used in the laboratory. Laboratories that determine microgram or smaller quantities of elements should incorporate safeguards to prevent sample contamination from wall paints, work surfaces, floor cleaning compounds, etc.

6.2 *Insulation*:

6.2.1 Acoustical walls and ceilings are desirable for noise control and to eliminate or reduce potential hazards to hearing. The room noise level shall be reduced to or below the level required by applicable regulations.

6.2.2 The walls and ceilings should be heat-insulated so that an air-conditioning unit of minimum size satisfies the requirements.

6.2.3 The enclosure should be well sealed to prevent entrance of dust and corrosive materials.

6.3 *Air Conditioning*—An air-conditioned laboratory is necessary to ensure maximum precision of photographic and electronic recording instruments, but particularly for vacuum and air-path optical emission spectrometers. Low humidity will reduce absorption of water vapor by specimens that must be powdered and mixed homogeneously. A dry atmosphere will also reduce "spitting" and improve the burn in a d-c arc, or even a spark excitation, and reduce leakage of charge from the capacitors in the electronics of an optical emission spectrometer. The relative humidity shall be held constant at some value between 35 and 50 %, and, if possible, within ±1 %. For older instruments, it may be necessary to hold the temperature within ±0.5°C at some value between 21 and 24°C (70 and 75°F). For newer instruments, the recommendation of the manufacturer should be observed. The air-filtering system shall be maintained at designed efficiency to prevent contamination from adjacent laboratories.

6.4 *Vibration*—Vibration shall be minimized in spectrochemical laboratories and other support laboratories by good construction and by isolation from vibrating machinery. In addition, shock mounts and vibration dampeners may be required for analytical balances, microphotometers, spectrographs, and for optical and X-ray emission spectrometers.

6.5 *Darkroom*—Light-colored walls are recommended for the darkroom, which needs to be light-tight. A maze entrance painted with a flat black paint or weatherstripped door, or a revolving light-tight door will provide the necessary darkness. Fluorescent lights should not be used in a darkroom because of the afterglow. Baffled ventilation openings should be used. An exhaust blower is recommended if ammonia or organic solvents are used.

6.6 *Microphotometer Room*—The microphotometer is best operated in a room without direct sunlight, and where the artificial light may be dimmed according to the preference of the operator.

6.7 *Hoods*—The optical emission spectrograph or spectrometer, plasma spectrometer, atomic absorption spectrophotometer, and flame photometer all require small hoods with dampers to carry away toxic fumes. If high-temperature flames (for example, nitrous oxide) are employed, joints shall be welded and not soldered. The plasma spectrometer also requires small hoods to carry away excess heat generated by the plasma torch and the r-f generator.

7. Services

7.1 *Electrical Power*—Electrical power shall be supplied as determined from load requirements in accordance with the latest revision of the National Electrical Code. High-voltage leads shall be as short as possible and well protected. Voltage control should be maintained within ±2 % by motor-generator sets, or electronic or magnetic regulators, preferably in an area outside the laboratory. Provisions should be made for a large number of wall outlets; 120 and 208-V single-phase ac shall be available. Outlets for 208-V three-phase ac may be needed for motor-driven machines or r-f generators. Older equipment may, however, have been designed for 220-V three-phase ac. Therefore, the recommendations of the equipment manufacturers and local engineers should be followed in designing the electrical service for the laboratory.

7.1.1 A good r-f ground shall be provided between the source unit and the excitation stand of the optical emission spectrometer. Connect the spectrometers and read-out units to this with copper braid as prescribed in Practice E 528. The inductively coupled plasma r-f circuits should be well-shielded and properly grounded to comply with Federal Communications Commission rules and regulations regarding spurious emissions. The r-f ground should be made of 12.7-mm (½-in.) diameter copper tubing, should have no right-angle bends, should not be clamped or the surface obstructed in any way, and should end in a deep well that is below the water table. Additional details on r-f grounding may be found in Practice E 528. The optical emission spectrometer equipment shall be free of building grounds for safety and prevention of ground loops.

7.2 *Water*—Hot, cold, distilled, deionized, and refrigerated water are needed in the photographic processing room. Water is also required for cooling electrode stands and clamps, atomic absorption flameless atomizers, and the r-f coil of an inductively coupled plasma. Water may also be required for cooling samples on surface grinders, cut-off wheels, remelt furnaces, and for chemical facilities.

7.3 *Gas and Air*—Gas is required for the preparation of samples, unless another form of heating is supplied. Compressed air may be supplied from a mechanical pump equipped with a storage tank for constant pressure and with filters to remove water, dirt, oil, and grease, or from tanked compressed-air manifolds.

7.4 *Laboratory Gases*—Cylinder gases may be required for any spectrochemical equipment within the scope of this practice. Argon, helium, hydrogen, acetylene, nitrous oxide, nitrogen, propane, and mixed gases may be required. Sufficient space shall be provided for the cylinders, which shall be kept in a vertical position and always well secured. They shall not be used or stored near burners, hot plates, or in any area where the temperature exceeds 52°C (125°F). The contents shall be identified with labels or stencils, and color coding. This means of identification shall, under no circumstances, be removed from the cylinders.[4]

7.4.1 Two-stage regulators with pressure gages should be used as part of the basic flow system to deliver required cylinder gas to a particular instrument at a reduced pressure. Practice E 406 is specific with regard to the types of regulators, flow-metering valves, flow indicators, and tubing for gas transport that should be used and should be consulted when designing a gas-delivery system.

7.4.2 When used in the laboratory, portable cylinders of flammable gases such as hydrogen, ethylene, or acetylene shall be limited to a one-day supply or one cylinder. Flammable gases shall be physically separated from high-level oxidizing sources.

7.4.3 Reserve gas cylinders should not be stored in the laboratory area. Gas storage areas shall be adequately ventilated, fire resistant, located away from sources of ignition or excessive heat, and dry. All cylinders shall be chained in place or placed in partitioned cells to prevent them from falling over.[4] Flammable gas cylinders shall be stored separately from cylinders containing other compressed gases. In all cases, storage areas shall comply with local, state, and municipal requirements as well as with the standards of the Compressed Gas Association and the National Fire Protection Association. Access to gas storage areas should be limited to authorized personnel.

7.4.4 Permanent tank installations shall be located outside the building, away from combustibles, and shall be provided with natural or forced ventilation, relief valves and piping, etc., as required by applicable regulations and by existing consensus standards.

7.4.5 Argon for controlled excitation should be stored as the liquid in large storage tanks of 64 000-ft^3 (1810-m^3) capacity. Small portable tanks may be easily contaminated by the supplier while being filled with argon. The usual contaminants are moisture, air, and carbonaceous gases, which give false results when using a vacuum spectrometer. Additional details with regard to the location of large storage tanks, the type of argon delivery lines, and the precautions to be observed are given in Practice E 406.

[4] Braker, W., and Mossman, A. L., *Matheson Gas Data Book*, Matheson Gas Products, East Rutherford, NJ, 1971, pp. VII–VIII.

8. Safety Precautions

8.1 *Chemical*—Precautions against chemical hazards shall be observed when chemical operations are involved. Refer to the Hazards section of Practices E 50.

8.1.1 *Flammable Storage*—Certain methods may require flammable chemicals. The storage area for flammable chemicals may be under the workbench or draft hood and shall be constructed of noncombustible material, shall be free of ignition sources, and shall be positively ventilated. The quantities of flammable chemicals and solvents stored in the laboratory, as well as the containers in which they are stored, shall be in compliance with Section 1910.106 of the rules and regulations of the Occupational Safety and Health Act.[5]

8.1.2 Chemical waste from laboratory operations may be flammable, toxic, noxious, or a combination of these. Combustible waste materials and residues in the laboratory shall be kept to a minimum, stored in closed metal waste cans, and disposed of daily or as required. Proper planning will consider safe collection, storage, handling, and disposal of these wastes to ensure safety and prevent pollution.

8.1.3 Personnel who use cylinder gases shall be thoroughly trained to observe precautions when handling gases. All laboratory and stockroom personnel shall be thoroughly trained in the proper procedures for transporting gas cylinders. They shall be instructed on how to identify compressed gases. All personnel should be familiar with the proper methods for securing cylinders and the correct procedures for attaching pressure regulators, flowmeters, or flow lines to an instrument. Refer to the Significance and Use section of Practice E 406 for additional information on the use and handling of cylinder gases.

8.2 *Electrical*:

8.2.1 All outlets and equipment shall be well grounded for safety and satisfactory operation of the equipment. Electrical shielding of X-ray and spectrographic equipment is required to prevent r-f interference and erroneous results. For an optical emission spectrometric power source or r-f generator, an r-f ground is recommended as prescribed in Practice E 528.

8.2.2 If high-voltage sources are used, it is recommended that all high-voltage leads have a 3-in. (76-mm) air clearance for 35 000 V, and any busses passing through walls should be protected by borosilicate glass insulation or its equivalent. Adequate grounding of circuits is vital. Electrode stands or clamps, or both, shall be surrounded by a well-grounded enclosure to protect the operator. If multiple-electrode stands of the oscillating or rotating types are used, all sections must be properly insulated from where the electrical discharge is taking place. If cooling water is recirculated from one holder to another, water with low mineral content must be used.

8.2.3 Equipment controls shall comply with applicable regulations and American National Standards Institute consensus standards, whichever provide greater personnel safety. As a minimum, each piece of electrical equipment shall have an emergency stop and a disconnect switch. Electrical control panels shall be interlocked. Where test access is required with power on, the test circuits shall be brought out to plug in receptacles at the panel box or cover. It is strongly recommended that ground-fault interrupt circuit breakers be installed wherever personnel work with line electrical circuits. When testing of spectrochemical equipment with circuits energized is required, personnel *must* be thoroughly familiar with the equipment and circuits.

8.3 *Mechanical*—Safety precautions pertaining to the use of sample-preparation machines, such as grinders and lathes, should be called to the attention of the laboratory operator. Special note shall be made concerning the use of goggles to protect the eyes and the use of an exhaust system in connection with grinders. Wearing of goggles near wheels, shields for grinders, and guards for belts and pulleys shall be required. Occupational Safety and Health Act rules and regulation, Sections 1910.132 and 1910.133, are specific with regard to eye and face protection and shall be strictly enforced.[5] In areas where the noise level may be high, the regulations outlined in Section 1910.95 of the Occupational Safety and Health Act rules and regulations shall apply.[5]

8.4 *Radiation*:

8.4.1 Colored glass or other ultraviolet-absorbing materials provide adequate protection from ultraviolet radiation emitted by an arc, nitrous oxide, or high-temperature flames, hydrogen or deuterium lamps, and inductively coupled plasmas. Suitable protection for eyes and skin shall be provided whenever lasers or mercury vapor lamps are used in the open.

8.4.2 X-ray equipment shall be operated with precautions against radiation exposure to personnel. Calibrated survey meters shall be used periodically to map the areas of the instrument, check shields, monitor beams, and to assure that the accepted radiation tolerances are being met. Areas containing sources of radiation shall be appropriately marked with standard warning posters. Film badges, pocket dosimeters, or film ring monitoring systems shall be used to gather radiation exposure data for permanent records on laboratory personnel in the room. Section 1910.96 of the Occupational Safety and Health Act on ionizing radiation shall be observed.[5] It is also recommended that all laboratory and maintenance personnel follow the safety procedures given in handbooks and publications from the National Institute of Standards and Technology[6] and the U. S. Government Printing Office,[7,8] or similar handbooks on radiation.

8.5 *Fire Control*—Fire control should be based on the recommendations of fire protection engineers. The selection and location of fire extinguishers requires a careful assessment of the control agent, the combustible material, and the personnel. All personnel should be instructed in the proper use of fire extinguishers located in the laboratory. All laboratory personnel shall be aware of potential fire hazards when working with

[5] *Federal Register*, Vol 44, No. 29, Feb. 9, 1979, Part II, or the latest issue thereof.

[6] NBS Handbook, *X-ray Protection*, HB 76, and NBS Handbook 111, ANSI N43.2-1971, National Institute of Standards and Technology, Gaithersburg, MD 20899.

[7] *Radiation Safety Recommendations for X-ray Diffraction and Spectrographic Equipment*, #MORP 68-14, 1968, available from U. S. Department of Health, Education, and Welfare, Rockville, MD 20850.

[8] U. S. Government Handbook 93, *Safety Standards for Non-Medical X-ray and Sealed Gamma-Ray Sources*, Part 1, General, Superintendent of Documents, U. S. Government Printing Office, Washington, DC 22025.

combustible materials, flammable chemicals, solvents, and gases. The local fire departments, including plant and municipal, should be apprised of the particular combustible materials, flammable chemicals, solvents, and gases used in the laboratory.

8.6 *Fumes and Dust*—All personnel should be aware of the potential health, fire, and explosion hazards from dust and fumes, which could be present in the spectrochemical laboratory. Sections 1910.93 and 1910.94 of the Occupational Safety and Health Act rules and regulations[5] should be referred to when determining the allowable limits of air contaminants and the type of ventilation required to reduce health and fire hazards.

8.7 *Radioactive Materials*:

8.7.1 When radioactive materials are handled, it is essential that the operator be acquainted with the dangers and safety precautions, and follow prescribed rules as designated by the Nuclear Regulatory Commission license and approved by the radiological safety officer. Two types of dangers exist: (*1*) irradiation of the body by external sources, and (*2*) inhalation or ingestion of radioactive particles. Of the two types, the second is more insidious and dangerous because the radiation in the immediate environs of the particle is intense due to the confinement of the particles. Radiation damage to body tissue may continue for a prolonged period because of the difficulty in eliminating radioactive particles from the body. Parts c and d of Section 1910.96 of the Occupational Safety and Health Act[5] should be consulted for additional guidance in handling radioactive materials.

8.7.2 Radioactive materials shall be stored separately from other laboratory reagents and equipment. They shall be stored in accordance with the Nuclear Regulatory Commission rules and regulations. Parts e, i, and j of Section 1910.96 of the Occupational Safety and Health Act are specific with regard to the identification of storage areas, storage of radioactive materials, and instruction of personnel working in these areas.[5]

8.8 *First Aid*—Occasional accidents or other hazardous occurrences may cause serious injury which would require the use of first aid to prevent further injury or to sustain life until skilled medical personnel arrive to begin treatment. Every spectrochemical laboratory and sample preparation room should have a first aid policy that conforms to company regulations or Section 1910.151 of the Occupational Safety and Health Act.[5] If company policy permits a first aid kit to be located in the laboratory area, all laboratory personnel should be aware of its location. The contents of each first aid kit should have the approval of local medical authorities and should be inspected annually to ensure that all material is in good condition and that perishable items are replaced.

8.9 *Personnel*—Personnel shall not be allowed to work in the laboratory until thoroughly familiar with the equipment and safety precautions. When repairs or modifications are being made to equipment, as in all laboratory operations, a second person shall be present at all times. In conformance with Section 1910.151 of the Occupational Safety and Health Act[5] and when company policy permits, supervisory or key personnel should receive periodic training in first aid. Special attention should be given to the treatment of injuries that would be unique to a spectrochemical laboratory, that is, chemical poisoning and burns, respiratory failure due to chemical fumes, electrical shock, etc. Each laboratory should have an emergency plan for the evacuation of personnel in need of hospital treatment (accident victims or individuals who become critically ill). All personnel should be aware of the type of transportation to be used and the location of the hospital to which the patient is to be transported.

8.9.1 Medical personnel should be informed of materials being processed, and medical monitoring of laboratory personnel is recommended. Medical personnel should also be informed of the type of injuries that can occur, such as those mentioned in 8.9, so that proper antidotes, medications, and equipment will be available.

9. Keywords

9.1 safety; spectrochemical analysis; spectrographic analysis