ITEM 5
Propellant Production

Propellant Production

Production technology, or "production equipment" (including its specially designed components) for:
(a) Production, handling or acceptance testing of liquid propellants or propellant constituents described in Item 4.

Nature and Purpose: Individual components of liquid propellant production equipment are common to any petroleum distillation facility or large chemical plant. Typical components include reactor tanks, condensers, recovery columns, heaters, evaporators, filter assemblies, decanters, chillers, gas separators, and centrifugal pumps. None of these components by themselves is specially designed for use in making liquid propellants. However, when combined into a propellant production facility, such a facility is generally optimized for the production of a particular propellant and ill suited for making anything else.

The technologies for making liquid propellants are generally well known, although various companies may have proprietary procedures for maximizing yield, minimizing cost, or finding alternative uses for byproducts. Exceptions to this general rule include chlorine pentafluoride (ClF_5) and fluorox (ClF_3O), the manufacturing methods of which are closely held.

Acceptance testing of liquid propellants requires analytical equipment common to most chemical quality control labs, including equipment such as gas chromatographs, atomic absorption spectrometers, infrared spectrometers, and bomb calorimeters. This equipment generally can be used without modification to analyze liquid rocket propellants for acceptance.

Method of Operation: Specific production methods depend on the propellant being manufactured. Many of the constituents used in propellants are commonly produced for commercial purposes but require additional processing to purify, stabilize, inhibit, or blend to achieve certain properties. For example, sulfuric acid or magnesium carbonate is used to purify nitric acid. Commercial nitric acid, usually combined with water as hydrate, contains only 55 to 70 percent acid. Chemical processing is needed to break the hydrates to produce 97 to 99 percent pure, anhydrous (waterless) nitric acid. To form Inhibited Red Fuming Nitric Acid (IRFNA), N_2O_4 is added to the concentrated nitric acid to stabilize it against rapid decomposition, and trace amounts of hydrogen fluoride (HF) are added to reduce corrosion of containers.

Typical Missile-Related Uses: Liquid propellant production and acceptance testing equipment are required to develop an indigenous capability to make propellants.

Other Uses: The equipment and technologies are in common use and widely known in the petroleum and chemical production industries.

Appearance (as manufactured): In general, complete liquid propellant manufacturing facilities are not bought and transferred in one piece; they are assembled from many common pieces of chemical and industrial process equipment. Unless a turnkey plant is shipped, the most likely encountered items are probably the plans, drawings, calculations, and equipment lists associated with a plant design. There is even commercially available software that assists chemical engineers in designing such facilities.

Appearance (as packaged): The size of liquid propellant manufacturing equipment dictates the packaging. Smaller machines are crated in shock-absorbing containers or attached to cushioned pallets isolated from other packages. Larger machines are disassembled for shipping and reassembled onsite, and their components are packaged separately in crates or pallets.

(b) Production, handling, mixing, curing, casting, pressing, machining, extruding or acceptance testing of solid propellants or propellant constituents described in Item 4 other than those described in 5(c).

Nature and Purpose: The production equipment and infrastructure necessary to produce solid rocket propellant are complex and specialized. Facilities and equipment are necessary for preparing the various propellant ingredients, mixing and handling the propellant, casting and curing the propellant inside the motor case, and other specialized operations such as pressing, machining, extruding, and acceptance testing.

Method of Operation: Solid propellant is produced by one of two processes, either batch mixing or continuous mixing. Most missile programs use the batch process to make solid rocket motor propellant. After receipt and acceptance testing of the individual ingredients, ammonium perchlorate (AP) and others are usually ground in a mill to obtain the required particle size. All ingredients, including the binder, AP, metal powder, stabilizers, curing agents, and burn-rate modifiers, are mixed in large mixers to form a viscous slurry. The propellant slurry is poured or cast into the rocket motor case, in which a mandrel creates a hollow chamber running down the center of the motor. The loaded motor case is placed in a large oven to cure the propellant. During curing, the slurry is transformed into a hard rubbery material, or propellant grain. The rocket motor with the cured propellant is then cooled, the mandrel removed, and any final trimming or machining operations done. Finished motors are usually X-rayed to ensure that the propellant grain is homogeneous, bonded everywhere to the case, and free of cracks.


Photo Credit: British Aerospace Limited

Figure 5-1: Tactical motors with cast-cure tooling attached.

In continuous mixing, the same propellant ingredients are continuously measured into a mixing chamber, mixed, and continuously discharged into the motor or other container until the required amount of propellant has been obtained. This type of mixing is difficult because it is hard to precisely measure small amounts of some ingredients such as curing agents required for some propellants. Continuous mixing is not, therefore, used to any large extent.

Typical Missile-Related Uses: Better solid propellants improve missile range and payload capability. Solid propellant production equipment and acceptance testing equipment are required for a nation to develop an indigenous capability to produce propellants for rocket-motor-powered missiles.

Other Uses: N/A.

Appearance (as manufactured): Specialized devices are used to cast propellant by creating a vacuum, which removes air from the propellant as the propellant is poured into the rocket motor case. The size of these devices varies with the size of the rocket motors, but principles of operation are the same. Cast-cure tooling for small tactical motors is shown in Figure 5-1. The equipment and process for a small motor are shown in Figure 5-2. The mixed propellant is poured from the mix bowl into a large casting funnel, which is attached to the rocket motor. A large valve in the neck of the casting funnel isolates the motor in the vacuum from ambient atmospheric con-

Photo Credit: British Aerospace Limited

Figure 5-2: Slurry cast double-base mixing equipment for a small motor.

Photo Credit: Thiokol Corp.

Figure 5-3: A propellant casting pit, with a motor case belowground.

ditions. Once the casting funnel is full of propellant, the valve is opened slowly to allow the propellant to flow into the rocket motor case. Very large motors are sometimes cast in a cast/cure pit, which is an underground concrete structure lined with heating coils. The entire pit is evacuated before casting operations start. A belowground casting/curing pit in operation is shown in Figure 5-3. As with other specialized propellant equipment, the casting equipment is generally constructed on-site; its size depends on the size of the motor and the manner in which the casting operation is done.

Curing equipment ranges in kind and size from large, electrically or steam-heated ovens to large, heated buildings. This equipment is not particularly specialized because the process is a simple one, requiring only that motor temperature be raised for a given amount of time. Large cast/cure pits are permanent, on-site facilities.

The equipment used for acceptance testing of a batch of propellant is identical to the equipment found in an analytical chemistry or a materials testing laboratory. This equipment is used to perform chemical testing to verify the composition; to burn small amounts of propellant or test subscale motors to verify burning rate; and to conduct tensile testing to ensure that the propellant has the physical properties required by the rocket motor design.

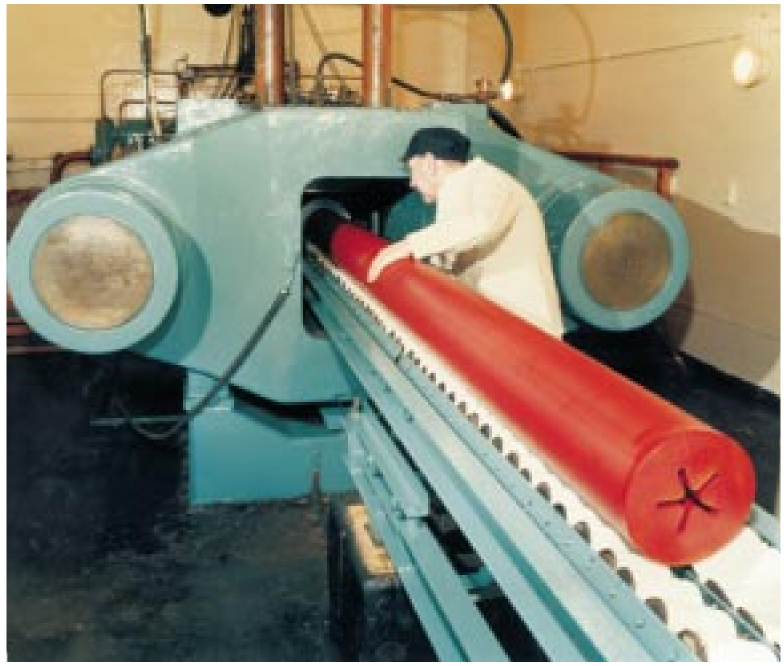
Photo Credit: British Aerospace Limited

Figure 5-4: A solid propellant extruder and grain.

Machining of solid propellant surfaces is generally done by large cutting machines specially modified to accommodate the safety hazards associated with solid propellants. Many of these types of machines are built specifically for a particular rocket motor.

Solid propellant grains for the large rocket motors of interest are usually too large to be directly handled by an extruder. However, some propellants of MTCR interest are extruded in a preliminary processing step. Extrusion is generally limited to propellant grains less than 0.3 m in diameter and has more application to tactical air-to-air, surface-to-air, and air-to-surface missiles. Such an extruder producing a relatively small grain is shown in Figure 5-4.

Appearance (as packaged): The size of solid propellant production equipment dictates their packaging. Smaller machines are crated in shock-absorbing containers or attached to cushioned pallets. Larger machines are disassembled for shipping and reassembled onsite. Their components are packaged separately in crates or on pallets.

(c) Equipment as follows:
- (1) Batch mixers with the provision for mixing under vacuum in the range of zero to 13.326 kPa and with temperature control capability of the mixing chamber and having;
  - (i) A total volumetric capacity of 110 litres or more; and
  - (ii) At least one mixing/kneading shaft mounted off centre;
- (2) Continuous mixers with provision for mixing under vacuum in the range of zero to 13.326 kPa and with temperature control capability of the mixing chamber and having;
  - (i) Two or more mixing/kneading shafts; and
  - (ii) Capability to open the mixing chamber;

Notes to Item 5:

(1) The only batch mixers, continuous mixers usable for solid propellants or propellants constituents in Item 4, and fluid energy mills specified in Item 5, are those specified in 5(c).

Photo Credit: Alliant TechSystems, Inc.

Figure 5-5: A 420-gallon propellant mixing bowl.

Nature and Purpose: Batch mixers are powerful mixing machines for batch quantities of very viscous material. They are derived from machines used to mix bread dough. Their purpose is to mix liquids and powders of differing densities into a uniform blend.

Continuous mixers are powerful mixing machines that operate in a flow through manner. They mix larger quantities than batch mixers for high volume production.

Method of Operation: Batch mixers operate much like a household electric mixer. The bowl holds the ingredients that may be added in sequence while the turning blades mix everything together. Temperature control and vacuum are maintained by surrounding the bowl with a water jacket and covering the bowl with a sealed lid.

Continuous mixers gradually feed all the ingredients simultaneously in their correct proportions through the mix region. The mixing/kneading shafts thoroughly mix the continuous flow of liquids and powders, and the uniform blend is gradually discharged out the large pipe in a steady viscous stream.

Typical Missile-Related Uses: Batch and continuous mixers are used to mix precise quantities of liquid propellant constituents and powdered propellant constituents into a very unifrom blend. This mixture will burn violently if ignited so safety procedures are critical. The blend produced is later cast and cured in another process to create a rubbery composite material that serves as the propellant in a solid rocket motor.

Other Uses: Batch and continuous mixers may be used whenever the production of a viscous blend is required. However, most commerical applications will not require the temperature control and vacuum capabilities specified in Item 5(c).

Appearance (as manufactured): The most distinctive components of a batch mixer are the mixing bowl and the mix blade assembly. The mixing bowls are typically 0.75 to 1.5 m deep and 1 to 2 m in diameter, as shown in Figure 5-5, but may be significantly larger for mixers greater than 450 gallons (1,700 l). They are double-wall constructed; the inner wall is made of highly polished stainless steel, and the outer wall is generally made of cold-rolled steel, sometimes painted. The space between the walls is used for a hot/cold water heating/cooling jacket. The outer wall has two valves for the connection of inlet/outlet water hoses, as shown in Figure 5-5. The bowl is generally welded to a thick steel rectangular plate with wheels at each corner. The wheels may have grooves so that the bowl assembly can be placed on rails for easier movement. Sometimes the upper rim of the bowl is a machined flat surface with a large groove to accommodate an O-ring (gasket); other times the mixer head is provided with one or more such grooves. The purpose of the O-ring is to provide a seal while the mixing operation is under vacuum. The blade assembly consists of two or three large blades, also made of highly polished stainless steel. Most assemblies use twisted-paddle blades, and one of the blades has an opening, as shown in Figure 5-6. Other assemblies use cork-screw-shaped blades. Although it is not evident in the shipping configuration, the blade assembly operates in a "planetary" manner; that is, the central blade rotates in a fixed position while the other one or two blades rotate about their own axes as well as rotating about the central fixed blade. The remaining mixer components include an electric motor, gear assembly, mixer head, and supporting structure.

Photo Credit: Thiokol Corp.

Figure 5-6: Open-blade configuration of a batch mixer.

Appearance (as packaged): Mixers may be shipped as complete units or as components. As precision-machined devices, mixer blades are packaged to protect them from damage and the elements. They are likely to be incorporated into the mixer head and frame assembly, and securely cradled in shock isolation material blocking during shipping. Mixing bowls are large, heavy pieces of equipment also likely to be shipped in large, strong, wooden crates. They are securely attached to the crates to avoid damage. Crates tend to lack any distinctive features or markings.

(3) Fluid energy mills usable for grinding or milling substances specified in Item 4;

Nature and Purpose: Fluid energy mills use high-pressure air to cause particles to rub against one another and thereby grind them down to very small particle sizes, generally 20 microns or less. Particle sizes in this range are not easily obtained by other grinding means such as hammer mills. Fluid energy

Produced by companies in

- China
- France
- Germany
- United Kingdom
- United States

mills are also much safer for grinding explosive materials such as Octogen (HMX) and Cyclonite (RDX).

Typical Missile-Related Uses: Fluid energy mills produce fine-grained AP, HMX, or RDX powders used as oxidizers or burn-rate modifiers for solid rocket fuel.

Other Uses: Fluid energy mills are also used in food, mining, and paint pigment industries.

Appearance (as manufactured): Fluid energy mills are extremely simple devices with no moving parts. Most are flat, cylindrical devices made of stainless steel and measuring 7 to 10 cm in height and 7 to 40 cm in diameter. They have an inlet and outlet port for the attachment of ancillary equipment. The interior of the mill is a tubular spiral that the material to be ground is forced through with high-pressure air. Particle size is controlled by the rate at which the material to be ground is fed in, and the air pressure or airflow rate. Three such mills are shown in Figure 5-7.

Appearance (as packaged): Fluid energy mills are generally shipped in wooden crates with foam or packing material used to protect them during shipment. The crates are not distinctive.

(4) Metal powder "production equipment" usable for the "production," in a controlled environment, of spherical or atomised materials specified in 4(b) (3) or 4(b) (4) including:
   - (i) Plasma generators (high frequency arc-jet) usable for obtaining sputtered or spherical metallic powders with organization of the process in an argon-water environment;
   - (ii) Electroburst equipment usable for obtaining sputtered or spherical metallic powders with organization of the process in an argon-water environment;
   - (iii) Equipment usable for the "production" of spherical aluminum powders by powdering a melt in an inert medium (e.g., nitrogen).

<u>Notes to Item 5:</u>

(2) Forms of metal powder "production equipment" not specified in 5(c) (4) are to be evaluated in accordance with 5(b).

Method of Operation: The most common approach for producing fine metal powders for use as constituents in missile propellants is the molten metal process using equipment specified in Item 5(c)(4)(iii) above. This process scales well and can be used to make large amounts of powdered metal cost effectively. Both the plasma generator and electroburst methods are relatively new in the application and are not in widespread use on production programs. They are currently considered laboratory or R&D processes when compared to the molten metal process.

Photo Credit: The Charles Stark Draper Laboratories, Inc.

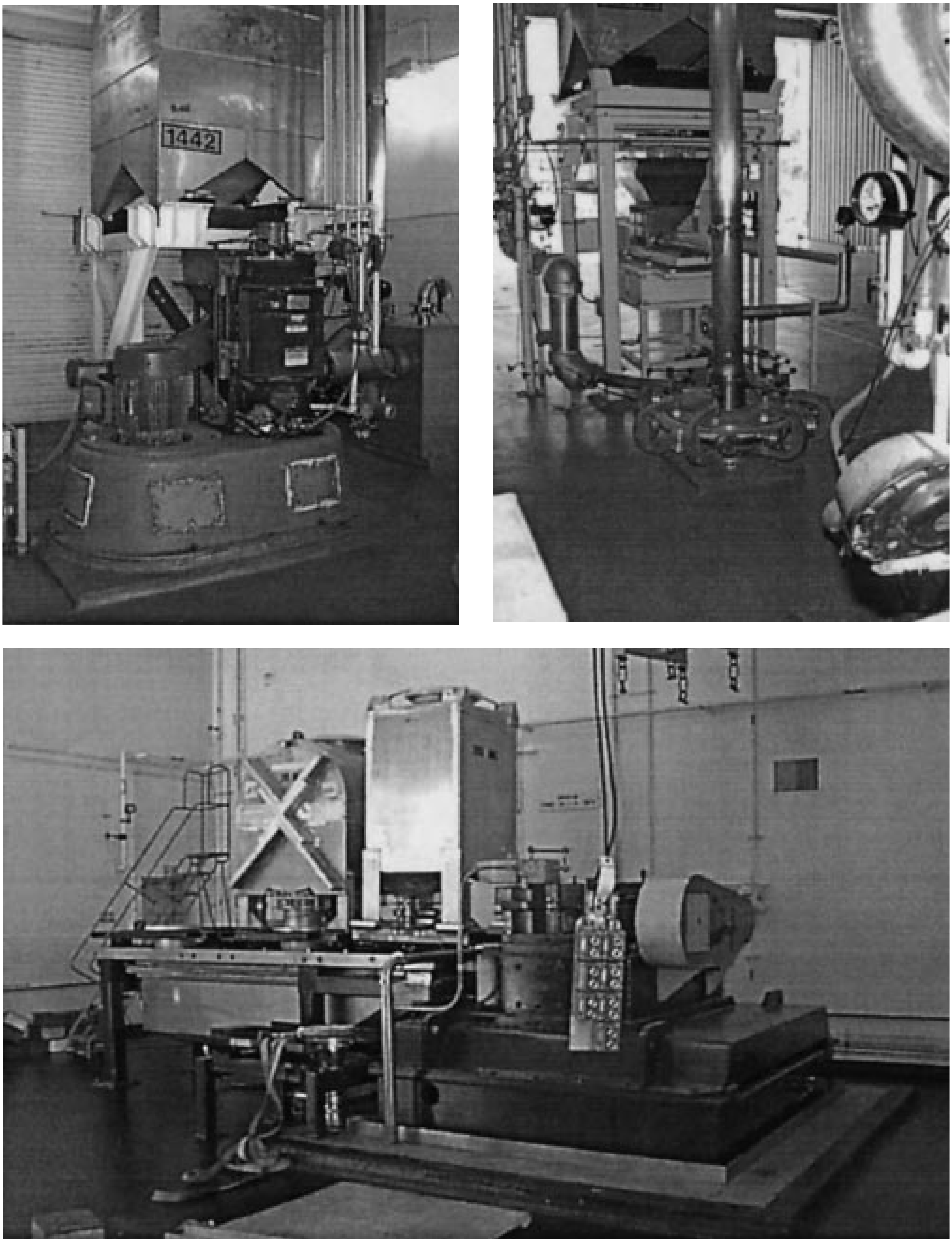

Figure 5-7: Three examples of fluid energy mills showing their various configurations.

In the molten metal process, molten metal is sprayed into the top of a large tank with an inert or controlled atmosphere. This atmosphere may be nitrogen, argon, or a mixture of both gases. A small controlled amount of oxygen is added to the atmosphere inside the tank when a thin oxide coating is desired on the metal powder. The molten metal spray causes small droplets to form, cool, and solidify into spherical particles of powder that fall to the bottom of the tank. The particle size of the metal powder is controlled by the size of the spray orifice.

Typical Missile-Related Uses: Atomized and spherical metallic powder production equipment is used to produce uniform, fine-grained metal powders used as a constituent in solid and liquid rocket fuel. Metallic powder is used to enhance the performance characteristics of the motor. Powdered metals are crucial in modern composite solid propellant motors. Atomized and spherical metallic powders in missile propellant increase missile range and payload capability.

Other Uses: Atomized and spherical metallic powder production equipment may be used to produce metal powders for many commercial applications, from pigments in metallic paints to fillers in structural adhesives.

Appearance (as manufactured): Equipment to produce atomized, spherical metal powder via the method described above is readily assembled from common equipment. The equipment includes a large tank into which the liquid metal is sprayed; a pump attached to the tank to remove the air; a filling system for the inert gas (e.g., tanks and a valve); a heater in which the metal is melted; and a sprayer-and-nozzle assembly that injects the metal into the tank.

Appearance (as packaged): An atomized-metal maker is not shipped as a single unit. Instead, its components are disassembled, packaged, and shipped like most industrial equipment. Smaller pieces are boxed or crated and secured to a pallet. The tank is boxed to protect it from denting. Spray nozzles are packaged separately in protected boxes.