AUTOMATIC CONTROL OF FOUNDRY
MOLDING SAND CONSTITUENTS

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Ву

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ABSTRACT

Some of the advantages and problems involved in the conversion from manual to automatic control of foundry molding sand constituents are discussed. The principal problem is that a transducer capable of monitoring each additive content is currently not available. The results of a search for an analytical technique suitable for such a transducer are presented. Of the numerous analytical methods reviewed, the multiparameter electrical technique, appeared to be most advantageous at the time that the survey was made. A description of its experimental evaluation is given. While a suitable transducer is not positively identified, guidelines for further work are suggested.

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TEXT

CHAPTER 1

INTRODUCTION

The green sand molding process is used extensively by the foundry industry to produce ferrous castings. cavity of the desired shape is formed in the sand with a pattern. Molten iron is poured into this cavity and allowed to solidify. Upon solidification the casting is broken out of the mold, allowed to cool, and then cleaned. Green sand molding is a very old production process. It has been dised by mankind for centuries. At first, sand that is now. classified as naturally bonded, was the only material used for green sand molding. In early times a successful foundry operation depended upon the availability of a sand deposit with a geological origin such that silica sand grains and clay binder existed in a suitable combination. Synthetically bonded sands were first introduced in the early 1900's, when a sand consisting of very pure silica sand grains was bonded with an appropriate quantity of fire clay. These sands , offered little improvement over the naturally bonded sands. A big improvement came when high strength bentonite clay was first used as a binder. The use of this material enabled a reduction in the binder content and improved many other sand properties. Nowadays most foundries use synthetic sands

using bentonite as a binder.

In addition to the sand grains and clay, a typical synthetic green sand also contains a small amount of water, called tempering water, and some finely ground bituminous coal, known as seacoal. The function of the tempering water is to make the sand-clay mixture somewhat plastic and to develop cohesive strength. Plasticity is required so that the sand can be molded according to the contours of a pattern even if it has deep draws. Strength is required so that the mold will retain its shape while the pattern is being drawn, handled physically during the core setting, mold closing, and during transfer to the pouring area operations, and while filling with molten iron. Seacoal is added to improve the surface finish of the casting. These materials and processes are described in more detail in the second chapter.

Heat, which transfers from the molten metal into the mold as the metal cools and solidifies, affects the molding sand in a number of ways, four of which are mentioned below.

Firstly, clay loses all its adhesive power after subjection to high temperature. According to Grim (1), this is a result of either vitrification or complete dehydration, both of which destroy the bonding property of clay permanently. Such material is termed "dead" clay in contrast to the "active" or "live" clay. This occurs only near the mold-

2

metal interface where temperatures are the highest. Clay some distance from this area is only partially dehydrated and can be re-vitalized with the addition of more tempering water.

Secondly, the heat turns the seacoal in a thin layer next to the mold-metal interface into coke which gives rise to a reducing atmosphere inside the mold cavity and closes in the openings between individual sand grains at the mold-metal interface. These two phenomena prevent the casting defects known to foundrymen as sand burn-on and metal penetration respectively. More detail is given in chapter two.

Thirdly, the heat drives some of the tempering water from the sand mold in the form of steam.

Fourthly, a layer of sand adheres to the raw casting when it is broken from the sand mold. This sand is lost during the cleaning process and represents a drain on the total amount of sand in the system.

These four effects along with the fact that in a modern mechanized foundry, sand is continously recycled, make it necessary to add new sand, clay, seacoal, and water to compensate for the depletion of existing constituents.

The total amount of each additive in the sand must be rigidly controlled or else the quality of the castings produced will suffer. Part of the third chapter of this report

deals with the required amount and allowable variation of each additive.

In most foundries the rate at which new additives are introduced is controlled manually by the operator of the sand mixer. This man has two basic sand testing methods which assist him. One is as old as the foundry industry The test, known as the hand squeeze method, simply involves picking up a handful of sand and squeezing it. experienced operator can determine its clay and moisture content from its texture, compactibility, fracture characteristics, etc. with a reasonable degree of accuracy. The hand squeeze method remains the front-line method of monitoring the constituents of foundry molding sand. second control is an entire battery of tests which have been developed to assist the muller (sand mixing machine) operator. Most foundries employ a man (or men) to perform these tests periodically and post the results near the muller In one typical automotive foundry, namely General operator. Motors of Canada Ltd. in St. Catharines/ Ontario, these tests The muller operator uses are done once every 30 minutes. the results obtained from these testing methods to monitor his hand squeeze technique and to control the seacoal content.

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This control procedure has several inherent weaknesses. First, the sand is tested at relatively lengthy intervals and long after it is mixed. Second, there is a high degree of

reliance on the human element. • The combination of these two factors result in a slow response to changes in the return sand analysis, and causes inconsistencies in the prepared sand additive content.

Some foundries have equipment to automatically control the sand moisture content. These controls appear to lack a certain amount of sophistication, as evidenced by the fact that, in several installations, they have been observed to be on manual over-ride on a continuous basis.

The ability to monitor the composition of molding sand and to control the addition of all the constituents automatically would be a significant improvement over the existing manual control procedures. Such an advance would constitute an improvement in the quality of castings produced as a result of a more consistently prepared sand mixture, and a potential labour savings. The General Motors foundry in St. Catharines, for example, has five separate sand preparation systems which, depending upon the demand for castings at any given time, require as many as fifteen muller operators and three sand analysts. A reliable control system on all five sand systems could reduce manpower requirements to as low as six men, assuming that two men on each of three shifts would be required, to service the control systems, realizing savings of up to \$274,560 annually (12 men X 2080 hours/year X \$11.00 /hour = \$274,560.00). The major problem in designing an automatic monitoring and control system, would be to obtain suitable transducers capable of detecting each additive at the required level of accuracy and repeatability. Such transducers are not used in existing foundry practice. Therefore the primary objective of this report is to review monitoring practice and establish some of the basic requirements for automatic control of foundry molding sand additives.

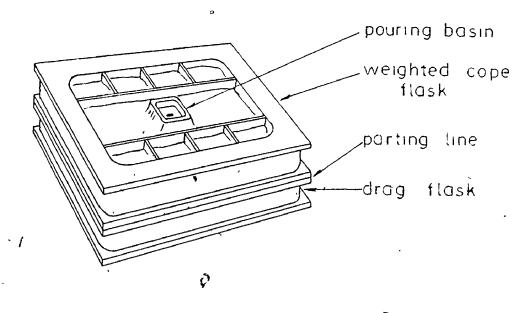
CHAPTER 2

THE FOUNDRY GREEN SAND SYSTEM AND MATERIALS

The green sand, cope and drag molding process is used to produce the bulk of all ferrous castings. According to Heine (2), it occupies this position because of its cost advantages, versatility, and adaptibility to automation.

A visitor to a typical mechanized foundry, which utilizes a green sand, cope and drag molding process in the production of grey, maleable, or nodular iron castings can expect to find several systems involved in the production process, each of which can, to a limited extent, be operated independently. First, there is the system to melt scrap steel, sprue returns, and pig iron into molten iron and prepare it for pouring. Second, there is a system to recondition and recycle green molding sand. The word green is applied to the molding sand of this particular process because it is used in a wet condition. The use of the term arose from the wood industry where wet wood is also described as being green. Third, there is a system of machines used to. manufacture cores which are required to produce a casting having internal bavities such as an automotive engine block. Cores are made from silica sand grains bonded together with

an organic resin. Fourth, there is a molding system that squeezes the green sand into molds, assembles the molds, takes them to the pouring area where they are filled with iron, and delivers them to the shakeout area after sufficient cooling time in the mold for solidification. Figure 2-1 shows that the mold is made in two halves. The bottom half is called the drag, and the top half is called the cope. Both are made by compacting green sand inside very rigid steel containers called flasks. External casting surfaces are formed by the impressions in the cope and drag parting faces, while internal casting surfaces are formed Iron is introduced into the mold through the pouring basin and runners. These are also filled with iron where the mold is poured. Fifth, the shakeout system separates castings and sprue (solidified pouring basin and runners) which at this point, are still red hot, from the sand in which they solidify. The castings and sprue are loaded into a bucket type conveyor where they cool to ambient temperature. The sand is returned into the sand system for recycling. Sixth, when the castings and sprue have cooled, they reach the finishing system. Here the castings and sprue are separated. The sprue is transported to the melt department where it also is recycled. castings are put through shot blast machines and tumbling mills to remove fins and sand aghering to the surfaces.



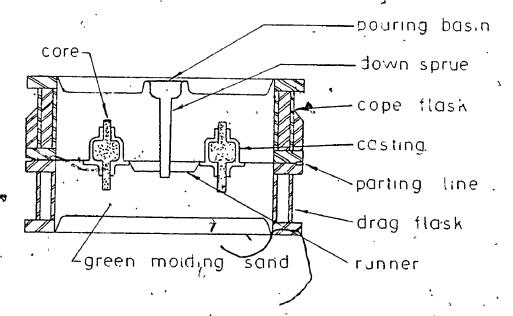


figure 2-1. Assembly and Cross-section of a Typical Green Sand Mold.

this point, subject to approval by the quality control group, the castings are ready for shipment.

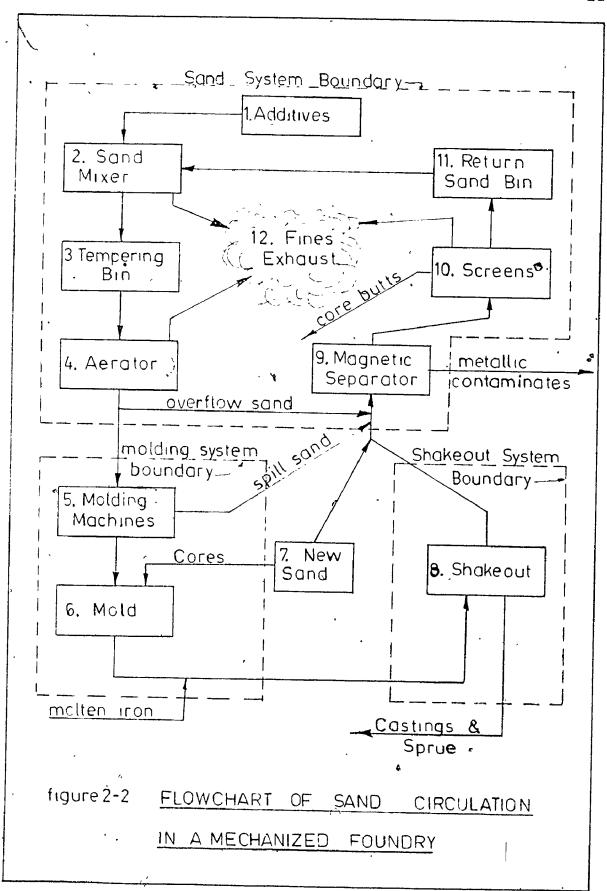
The above is a very brief picture of what the foundry production process is like. While it is beyond the scope of this report to describe all the systems mentioned above in great detail, it is in line with the objectives to describe the sand system operation and the constituents of green molding sand more closely.

A. The Sand System

The basic function of the sand system is to provide a supply of green sand, consistent in composition to the molding system. As mentioned previously the sand system reconditions and recycles the sand. It can be operated independently of the rest of the production process. The system with one exception, the muller, is made up entirely of material handling and storage components, namely belt conveyors, screw feeders, bucket type elevators, chutes, vibrating feeders, bins, tanks, and pipes. While the actual system configuration varies somewhat with each installation, the flow chart in figure 2-2 is generally applicable to all sand systems. A description of each numbered item on the flow chart is given below.

1) Additives

The additives used in the process of reconditioning green sand are water, clay and seacoal. As mentioned in the introduction, these must be added each time the sand is recycled to compensate for the depletion brought about by the heat of molten iron. The clay is actually a 30 - 70 combination of western bentonite and southern bentonite. These two clays are thought to be quite similar structurally but they bring out different mechanical properties in the green sand. Southern bentonite and water are mixed into a slurry which is then piped directly into the sand mixer. Western



bentonite and seacoal are fed into the sand mixer in dry form.

2) Sand Mixer

The sand mixer or muller blends the additives in with sand, which is continuously fed into the mixer from the return sand bin via a belt conveyor, in such a way as to obtain a uniform composition at the discharge door. As mentioned in the introduction, the rate at which additives are introduced into the sand is manually controlled by the muller operator.

According to the National Engineering Company (3), who are manufacturers of mulling equipment, the blending is accomplished "by the movement of the differently angled plows which divide and mix stratified charges of sand and by the smearing action of the rolling weighted wheels". It should be noted that the mixing is a physical action only. A chemical reaction is not involved. The current drawn by the muller electric motor is determined by the amount of sand in the muller and is regulated utilizing the muller discharge door. The control mode is simply on-off. When the current is below a predetermined level, the door is closed. When it reaches a higher predetermined level, the door opens, allowing sand to be discharged at a faster rate than it is fed into the muller. As a result the current falls off until the lower level is reached which cause the

door to close. The effect of this control action is to control the amount of sand in the muller. Upon being discharged from the muller the prepared sand is transported into the tempering bin.

3) Tempering Bin

Properties of green sand that are useful for molding purposes such as flowability and green compression strength improve if the sand is allowed to sit prior to use. The function of the tempering bins is to hold the prepared sand for a minimum of one hour to allow this improvement to occur. It should be noted that some foundries do not have tempering bins, claiming that the "cost of instalation does not justify the marginal improvement in sand properties". An example of a foundry that has adopted this line of thinking is the Chrysler Huber Avenue Foundry in Detroit Michigan. However, most foundrymen agree that the best practice is to use tempering bins. They also claim that the use of tempering bins greatly reduces the occurrence of a casting surface defect, known as scabbing, which is caused by the presence of unabsorbed or 'free water' in green sand.

4) Aerator

Sand emerges from the tempering bins in a heavy, lumpy condition. The aerator breaks up the lumps and fluffs up the sand such that it falls into the recesses of pattern more easily than in the lumpy condition. After leaving the

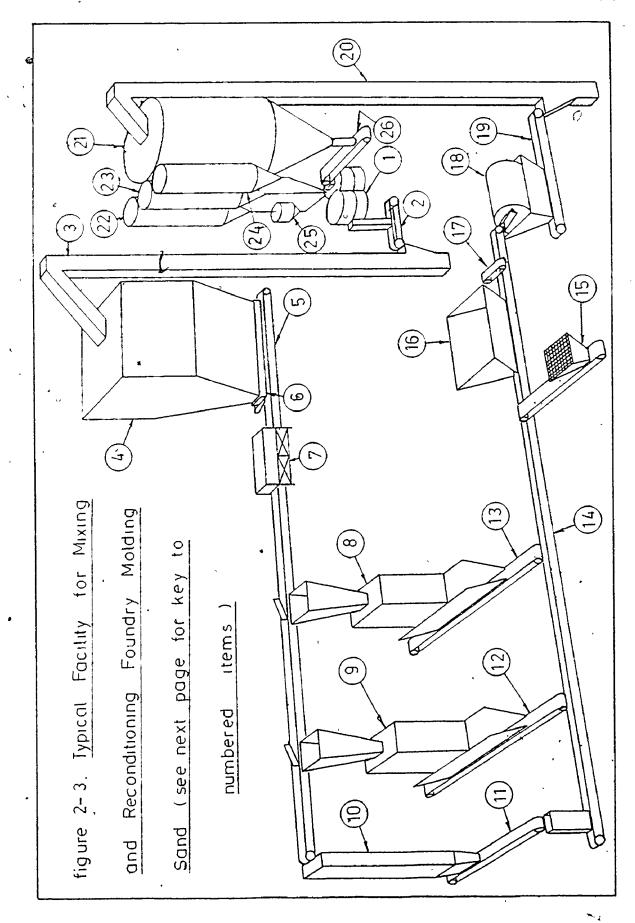
aerator most of the prepared green sand leaves the sand system and enters the molding system. This is illustrated on figure 2-3 which is a pictorial of a sand system. Sand entering the molding system is removed from the distribution belt at each molding machine by a plow. A smaller portion of the sand leaving the aerator remains in the sand system. It is transported to the return sand system via the overflow chute, a series of belt conveyors, and an elevator.

5) Molding Machines

Its worthy of note that an automated molding line usually has two molding machines; one to make the lower section of the mold known as the drag, the other to make the upper section of the mold known as the cope. Both the cope and drag are made by compressing a quantity of green sand against a pattern inside a rigid steel flask. A portion of the sand directed to the molding machines spills over the sides of the flask just prior to the squeeze cycle. This 'spill sand', like the overflow sand, is returned into the sand system via a series of conveyor belts and an elevator.

6) Mold

The copes and drags are generally assembled into molds on a pallet conveyor. The drags are placed directly on the pallets, cores are set into them if required, and the copes are placed on top. The mold conveyor passes through the pouring area where the molds are filled with molten iron.



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Key to numbered items in figure 2-3

number	item	
, 1	sand muller	
2	prepared sand belt	
3	prepared sand elevator	
4	tempering bin	•
5	distribution belt	
6	vibrating feeder	
7	areator	
8	drag molding machine	1
9	cope molding machine	}
10	overflow chute	
11	overflow belt	
12	cope spill sand belt	
13	drag spill sand bo lt	
14	return sand belt	
15	sand addition system	
16	shakeout section	
17	magnetic tramp iron separator	
18	rotary screen	`
19	return sand belt	
20	return sand elevator	
21	return sand storage bym ,	
22	seacoal storage bin	
23	western bentonite storage bin	
24	southern bentonite storage bin	
25	slurry tank	
26	return sand feeder belt	

From the pouring area the mold conveyor carries the molds to the shakeout system where they are removed from conveyor. The elapsed time for this part of the cycle is sufficient to allow the iron to cool to below its solidification temperature.

7) New Sand

As mentioned in the introduction, the additions of new sand is necessary because of losses which occur when some of the green sand adheres to the raw castings when they are removed from the molds. New sand is introduced into the system either through cores or by direct addition. Direct addition of sand is necessary only when the losses exceed the amount of new sand introduced by cores. In the case of core sand additions the heat of the molten iron breaks down the resin binder reducing the core to loose sand. This loose sand enters the sand system at the shakeout. If the core is large or has bulky sections, the heat may not be sufficient to break it down completely. The remaining lumps known as core butts are undesirable in molding sand.

8) Shakeout

At the shakeout the cope and drag sections of the molds are separated and the castings and the sand that made up the mold are dumped into a heavy duty vibrating feeder. The pan of this vibrating feeder has gratings that allow the sand to fall through onto a belt conveyor below. The

castings and sprue are loaded into a conveyor which transports them to the finishing system.

9) Magnetic Separator

Small pieces of iron in green sand will damage even a steel pattern. At the surface of the mold a piece of iron will cause a flaw in the finished casting. Hence it is necessary to remove these from the sand. Foundries use magnetic separators which are suspended over a belt conveyor for effective separation of tramp iron from the sand.

10) Screens

Screens are necessary to remove non-magnetic foreign material and core butts from the sand.

11) Return Sand Bin

After screening, the sand is stored in the return sand bin. This storage bin along with the tempering bin has enough capacity to hold all of the sand in the entire system including that in the molds.

12) Fines Exhaust

Green sand additives are fine powders which cause dusty conditions if they are not contained. To provide a tolerably clean working environment, air-borne particulate matter must be collected with a dust collection system at all transfer points. Some or all of these fines could be returned into the sand system if it is so desired.

It is worth noting that there are three events which occur within the molding system that upset control over the

composition of green sand at the muller. First, on certain occasions some molds are not filled with molten iron. happens when the molds are damaged physically or when the sand composition is known to be incorrect. Sometime later an amount of return sand fed into the muller will have more than the normal amount of additives in it. Second, on other occasions the patterns are changed and a production run of a different casting commences. This almost always means a change in the amount of molten iron poured which results in a different rate at which additives are depleted from the sand. The total weight of cores set into the mold will also change? in most cases, resulting in a different amount of new core sand in the return sand. All of the new core sand grains should receive a uniform clay-water coating and dispersion of seacoal on the first pass through the muller. these effects make it necessary to adjust the additive feeders when the return sand reaches the muller. Third, the return sand temperature fluctuates. The largest temperature variation is an increase which occurs at the beginning of a production period when the first sand cycled through the molding and shakeout systems reaches the muller. The temperature will also fluctuate at the beginning of the production run of a different casting if the pouring weight changes. Temperature variations change the amount of water required in the reconditioning process because evaporation losses increase at higher

return sand temperatures.

In the case of all three of these events the muller operator must notice the change in the return sand, assess the situation, and make an appropriate adjustment to the additive feeders in order to maintain control over the green sand.

Finally, a word concerning the size and capacity of a green sand system. As mentioned in the introduction, the General Motors St. Catharines Foundry has five separate sand systems having prepared sand delivery capacities ranging between 250 and 600 tons per hour. The combined total capacity for that particular foundry is 1950 tons per hour. The total amount of green sand in each of these systems range between 300 and 1000 tons. Because the quantity of sand processed is great, a failure in manual control is an expensive proposition.

B. Constituents of Green Molding Sand

The synthetic green sand used in automated cast iron foundries has been described in considerable detail by Grim (1), Heine, Loper, and Rosenthal (2), Yearley (4), and the American Foundryman's Society (5). Synthetic green sand is a mixture of at least four constituents, namely silica sand grains, clay, water, and some carbonaceous material which is usually seacoal. The picture is complicated by the presence of quantities of these constituents that have been decomposed

or altered in some way by the heat of the iron. The four principal constituents are discussed below.

1) Sand Grains

According to Heine, Loper, and Rosenthal (2), silica sand grains make up fifty to ninety-five percent of the total material in green sand. This amount varies from foundry to foundry primarily according to the type of production equipment utilized. Silica sand is used by virtue of its low cost, availability, and favourable refractory characteristics to provide bulk for the material in a green sand mold. The chemical composition, grain shape, grain size, and grain size distribution of silica sand is peculiar to the deposit from which it originates. Little or nothing can be done to change these artificially. Generally the purest silica sand, 99.8 percent siO2 or better, has superior refractory qualities and is best suited for green sand used to mold ' cast iron. Sand with this degree of chemical purity is thermally stable at temperatures in excess of the cast iron pouring temperature. This helps to ensure the dimensional accuracy of the finished casting. Sand grains are inert material in a mold: Under normal circumstances they do not enter into any chemical reactions.

The shape of sand grains may vary from almost true spheres to needle-like splinters. The optimum shape for green sand is somewhere between these extremes. The grains

should be generally round but have rough surfaces and sharp edges. The amount of clay required by a sand of favourably shaped grains to achieve specified bonding properties is less than the amount required by sands of smooth, rounded or needle shaped grains. The sand grain size and size distribution also influence the properties of molding sand, Molding sand with a fine grain size gives a casting a relatively smooth surface finish. This desirable feature is offset by the fact that the mold permeability or porosity between grains is reduced by a finer grain size. A green sand mold must be permeable so that air and generated gases and steam can escape when it is filled with molten Molding sands with a broad grain size distribution generally have greater bonding strength than sands with similar average grain size but more uniform size distribution. A common parameter used by foundries to describe molding sands is known as the grain fineness number. The number designates the average size as well as proportions of smaller and larger grains in the sand mixture. The number is calculated from the proportions of a sand sample retained on standard seives, each with a certain number of holes per linear inch. The method of determining this parameter has been outlined by Heine, Loper and Rosenthal (2).

According to Draper (6) silica sand grains possess marked surface activity. They develop surface charges of

varying intensity on different areas of the individual grains. Such charge variations occur because abrasion during mulling, handling, and compaction produce minute fractures and creates new surfaces where molecular structures are disrupted. The electrically charged areas become receptive to either the positive or negative poles of water molecules and relatively positive areas attract the negative side of the water films adsorbed on the clay platelets. This surface activity phenomenon helps to hold a clay-water coating on each molding sand grain. This in turn helps to promote the bonding action of molding sand.

2) Bonding Clay

Clay in conjunction with water acts as the binding medium in a green molding sand. Sufficient clay is required to provide an envelope around each sand grain to confer to optimum physical properties to the sand mix. If the clay content is too low, the sand grains are not completely coated and the molding sand will not have adequate strength. If the clay content is too high, the sand is difficult to handle in its loose state and has a very low permeability in its compacted state.

There are several chemically and structurally different clays which are used as bonding agents in green molding sand. These different clays all have their own peculiar properties which are advantageous in certain

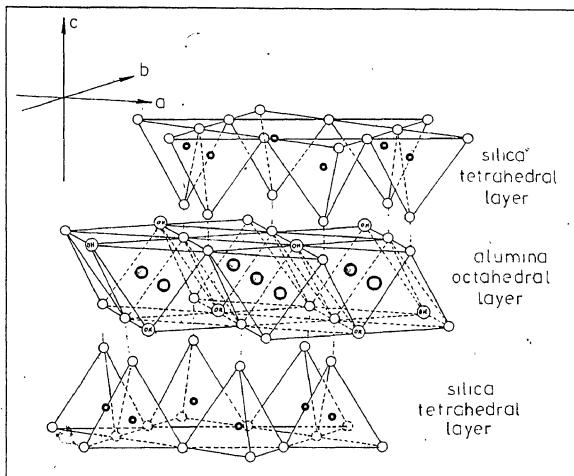
Asituations. For example, kaolinite, which is also known as fire clay, has a high refractoriness. According to Heine, Loper, and Rosenthal (2), refractoriness is the range of temperatures over which the material softens. Consequently the material is often used for cast steel molding where very high pouring temperatures are encountered. Bentonite, a clay with somewhat lower refractoriness but capable of developing stronger bonds when in the wet state, is used almost exclusively for cast iron molding sands. The discussion that follows is limited to this material.

Heine, Loper, and Rosenthal (2) report that bentonite is itself made up of several constituents. Montmorillonite is the principal constituent composing 85 to 90 percent of the clay. This constituent is responsible for the bonding characteristics of clay. Other constituents consist of finely divided quartz, silica, mica, and other minerals.

These other constituents are considered to be impurities and are difficult or impossible to separate from the montmorillonite. Montmorillonite is made up of extremely small flake-shaped particles. In the literature these particles are often referred to as platelets. The size has been estimated to be between 10 and 100 A thick and up to 3000 A in each of the other directions. Montmorillonite is one of a class of disilicate minerals which has a three layer sandwich construction. Figure 2-4 is a sketch showing

The two outer layers are made up of silica its structure. tetrahedra which may be represented by (Si₂O₅)²⁻. These tetrahedra form a hexagonal grid extending in both lateral directions. The tips of the tetrahedra all point in toward the sandwich layer which is made up of alumina octahedra. The octahedral layer may be represented by Al (OH) 3. tetrahedral and octahedral layers are combined so that the tips of the tetrahedra of each silica layer and one of the hydroxyl sheets of the octahedra layer are common. atoms common to both the tetrahedral and octahedral layer are oxygenan In figure 2-4 the common oxygen atoms are joined by a dotted line. These three layer units extend laterally in the a and b directions and are stacked one on top of another in the c direction. In the stacking of the silica-alumina-silica oxygen layers of each unit are adjacent to oxygen layers of the neighboring units with the consequence that there is only a weak bond between them. Water enters between the unit layers of the clay thus activating its bonding properties. The entry of water between the unit layers causes the clay to swell. Grim (4) reports that the theoretical formula for the structure given above is

(OH)₄ Si_s Al₄ O₂₀ n (interlayer) H₂O. Montmorillonite always differs from the theoretical formula because of substitutions of aluminum for silican in the tetrahedral layer and/or magnesium, iron, zinc, nickel, lithium, etc. for aluminum



- o oxygen atoms
- silicon and occasionally aluminum atoms
- o aluminum and sometimes iron or magnesium atoms fig 2-4. A sketch illustrating the structure of montmorillonite. The atoms joined by a dotted line are actually one, shared by both the octahedral and tetrahedral layers. The three layer structure is repetitive in the "c" direction up to about 10 thicknesses. It extends in the "a" and "b" directions such that the areal dimensions are between 10 and 100 times the thickness.

in the octahedral layer. These substitutions always result in electrical unbalance within the structure. Grim (1) states that many analyses have shown that the same net positive charge deficiency results regardless of the nature of the substitutions. The positive charge deficiency is offset by an appropriate number of cations which are held around the outside of the silica-alumina-silica units.

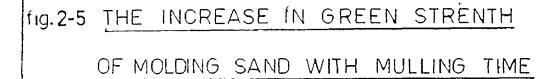
The type of ions held by montmorillonite influence the overall physical properties of the clay. The montmorillonite constituents of bentonite mixed in the state of Wyoming and Mississippi, U.S.A., hold sodium and calcium cations respectively. Sand bonded with the Wyoming or western bentonite is more difficult to handle when loose, has a lower strength when compacted, and has a greater tendency to swell with the addition of water than sand bonded with southern bentonite. Stephens and Waterworth (7) have investigated the physical property differences which may be attributed to the typed of cation held by the bentonite. This particular aspect of clay mineralogy is outside the scope of this report.

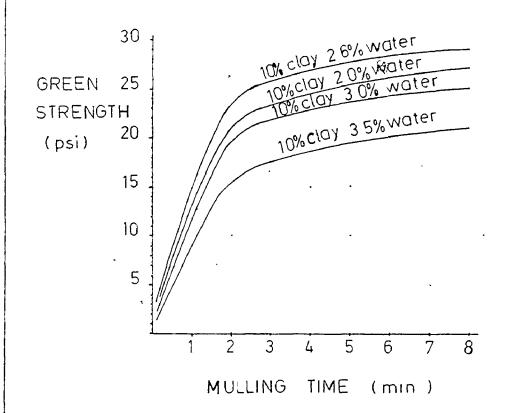
As mentioned in the introduction of this report, bentonite loses all its bonding capability when subjected to temperatures as high as the pouring temperature of iron. The resulting dead clay contaminates the sand if it accumulates. In actual practice much of the dead clay

leaves the system with the sand clinging to the rough casting and it remains at a tolerable level (see figure 2-2). Clay can be "deadened" in one of two ways. If heated in excess of its softening point (1800°F +, depending upon its origin), bentonite vitrifies and is structurally destroyed. If bentonite is heated to elevated temperatures below its softening point, all the water separating the silica-alumina-silica structural units is driven off. Once this happens, the bonding properties are destroyed permanently because water cannot be induced to enter the clay again. Thus new clay is required to coat the core sand which enters the sand system at the shakeout as well as to compensate for the clay "deadened" by the heat of the castings.

The direct measurement of clay content (active or dead) quickly, presents a difficult problem. Consequently it is usually obtained from a parameter refered to as green strength in the foundry industry. Green strength is expressed in pounds per square inch required to crush a standard specimen. The clay content is assumed to be directly proportional to the green strength. Determination of clay content in this manner is at best a poor estimate. Examination of figure 2-5 will clearly indicate that the green strength is dependent upon water content and mulling time as well as the clay content. Clay content is estimated in

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NOTE:

The values of maximum green strength are taken from RE GRIM, APPLIED CLAY MINERALOGY, McGRAW-HILL, 1962, fig 4-3, page 149.(1) The shape of the curves was adapted from DOYLE, MORRIS, LEACH, SCHRADER, MANUFACTURING PROCESSES AND MATERIALS FOR ENGINEERS, PRENTICE-HALL, INC. 1964, fig 8-39, page 153.(8)

this way because it can be done quickly.

to determine clay content. They range from exact to crude in accuracy. Some of these methods are described below. The American Foundryman's Society (5) defines clay in molding sand as "particles which fail to settle one inch per minute when suspended in water. These are usually less than 20 microns, or 0.0008 in., in diameter." accordance with this definition, one clay analysis method widely used by the foundry industry, is to agitate a sample of dried molding sand in a caustic soda solution and after a prescribed settling time siphoning off the portion in which the clay is suspended. The agitation, settling and siphoning procedure is repeated enough times until the water is clear after standing for five minutes. The remaining material is dried and weighed. The percent weight loss is called the AFS clay content. This would include all very fine material including silica fines, active clay, dead clay, and seacoal fines.

There are also a number of laboratory techniques

b) The primary drawback of the previous test is that all fines below about 20 microns in diameter are included in the clay content. The primary concern is the active clay content. There are at least two methods by which active clay content can be measured. These are the methylene blue dye absorption test and the nickel sulphate method. These analysis methods

have been described by Yearley (10).

c) There are several methods which correlate the active clay content to the compaction characteristics and strength of green sand. These methods, while they are very convenient to use, can be misleading. Large errors can be introduced if the mulling time and moisture content are not kept at consistent levels.

3) Water

Water activates the cohesive properties of clay in molding sand causing the mixture to develop strength and plasticity. As mentioned previously, water is absorbed by clay entering between and surrounding the silica-aluminasilica structural units. Draper (6) has reported that the absorbed water appears to be held in a definite immobile form in the clay without dissociating chemically. Clay is capable of absorbing water only up to a limited amount. The absorbed water is known as tempering water to foundrymen. Only tempering water is effective in developing the strength of molding sand. Any water in excess of the limiting amount is termed free water. The presence of free water in molding sand is undesirable. It reduces its green strength, makes it sticky and difficult to handle, and causes casting defects such as porosity and scabs. A scab is a surface defect that can be rectified but sometimes at greater expense than the casting value.

The water content of green sand must be within close tolerances in order to produce good quality castings.

Several different methods to measure water content have been developed to assist foundrymen in controlling this important variable.

- a) The standard method is to oven dry a molding sand sample to a constant weight at approximately 105 to 110°C. The weight loss is assumed to be the weight of water in the original sample. The water content is easily calculated once this information is obtained. This method, while accurate, takes at least an hour and not suitable for routine foundry use for this reason. Other methods produce quicker, though less accurate, results.
- b) A second method again involves calculation of the water content from the weight loss of a sand sample. The difference is that a stream of heated air is forced through the sample. Apparatus for this kind of a test can be purchased that will provide results in two to five minutes.
- c) A third method involves mixing a sample of molding sand with calcium carbide powder. The chemical affinity of this material for water results in the generation of acetylene gas. If the molding sand samples and the calcium carbide powder addition are always the same weight then the amount of acetylene gas generated is proportional to the water content. Standard apparatus for this kind of a test can be purchased

off the shelf. This is an excellent method for sands that contain volatile or oxidizable constituents.

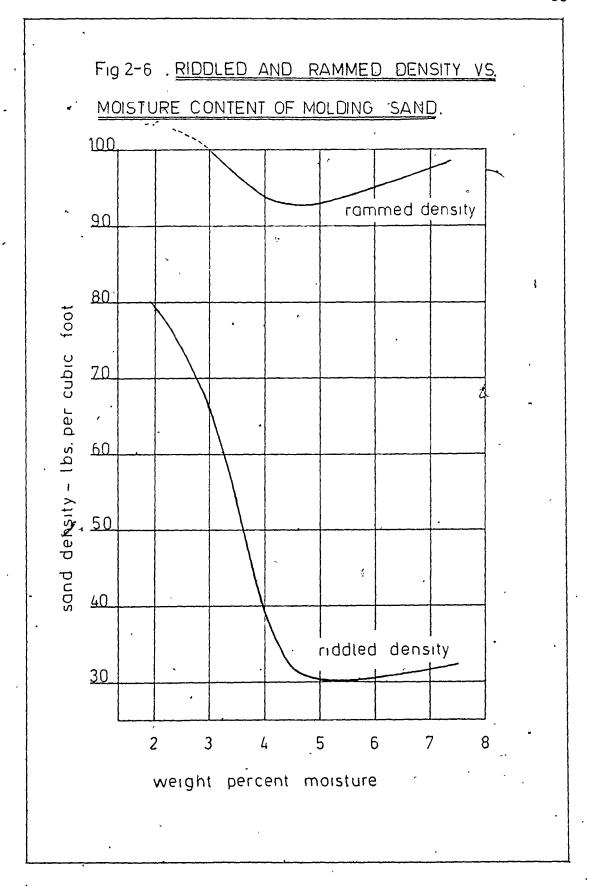
A survey of methods that could be used to monitor sand constituents automatically is presented in chapter four of this report. The importance of measuring the sand density is mentioned in the survey. Figure 2-6 illustrates the range of values that the density of a sand with a fixed clay content could assume, depending upon its moisture content and the way it was handled. The curves for a molding sand with a different clay content would be similar in shape but have different values.

4) Seacoal

Seacoal is a finely ground bituminous coal used in molding sands for cast iron principally for improving the surface finish and ease of cleaning the castings. The seacoal content varies from two to eight percent depending upon the application.

Seasoal is included in molding sand to prevent two distinct defects from occuring. These defects, known as sand burn-on and metal penetration to foundrymen, and the function of seasoal in their prevention, have been described by Thorpe (9), Yearley (11), and others.

a) Sand burn-on is a chemical bond between the casting and the mold. The bond is caused by the products of certain chemical reactions that occur as the molten iron fills the



mold cavity. The principal damaging product is FeO which is capable of dissolving the silica sand grains. FeO forms when molten iron combines with oxygen, present in the mold cavity prior to the pouring operation, and with water in the actual mold. Sand burn-on is recognized by its rust brown colour. Sand burn-on must be removed from the areas of a casting that require machining because it will damage the tooling.

b) Metal penetration is the result of liquid metal entering the voids in the sand mold and on solidification mechanically locking the sand grains to the casting surface.

The beneficial effect of seacoal is mechanical in nature. When the mold is filled with molten iron, the seacoal at and near the mold-metal interface is heated to temperatures substantially beyond its melting point. The seacoal melts and thoroughly coats the sand grains. This coating is insoluble to FeO. In fact it will reduce FeO back to iron. This prevents the occurence of sand burn-on. In addition to coating the silica sand grains the seacoal is actually changed into coke, swelling to between six and eight times its original volume and liberating a substantial quantity of gas. When the seacoal swells, the voids between the sand grains are filled. This prevents metal penetration from occuring. The gas liberated by seacoal is an undesirable element. It has been known to cause casting porosity if the seacoal content is too high. On the other hand not enough seacoal in

molding sand can result in sand burn-on and/or metal penetration. Experience at General Motors in St. Catharines has indicated that the optimum seacoal content varies from casting to casting. Good results are obtained on many castings if it is maintained between 4.7 and 5.3 weight percent.

Seacoal, as mentioned previously, is a finely ground bituminous coal. Because coal comes in numerous different grades, it is important for a foundry to specify its seacoal by either a proximate or ultimate analysis conventionally used for coal. Use of seacoal made from different grades of bituminous coal would affect the quality of the casting. An example of a proximate and an ultimate analysis is given in figure 2-7.

The seacoal content of molding sand is usually measured by heating a sample to 1800°F and holding it at that temperature until its weight is fixed. The weight loss is due to the volatile constituents of seacoal being driven off. The weight loss is directly proportional to the seacoal content.

C. Summary

The green molding sand preparation and reconditioning equipment, ingredients, and laboratory testing procedures
utilized by a modern cast iron foundry are described in some
detail in this chapter. The function of the muller operator

fig. 2-7 Typical Seacoal Analysis

Proximate analysis (moisture free)

volatile matter (VCM) 36 to 40 % fixed carbon (FC). 55 to 60 % ash 3 to 5 %

<u>Ultimate analysis</u> (moisture free)

H 5.6%

C 80.0 to 85.0%

N 1.0 to 3.0 %

O 6.0 to 8.0 %

S 0.8 % max.

ash 3.0 to 5.0 %

ash fusion point 2780 F

in maintaining control over the constituents is outlined in the introduction and elaborated upon in this chapter. The origin and nature of disturbances in the return sand, which upset control over the consistency of the prepared green sand is also discussed. Knowledge of all these things is essential to implement an automatic system to control the addition of green sand constituents.

CHAPTER 3

PRELIMINARY SPECIFICATIONS FOR AN AUTOMATIC SYSTEM TO CONTROL GREEN SAND ADDITIVES

Coughanowr and Koppel (12) have said that the practice of process control has been built largely upon the results of past experience and simple tests and, to some extent, upon intuition. However as indicated by Considine and Ross (13) the techniques of applying automatic controls to production processes are evolving into a mathematical science. Automatic process controls are widely used in many industries. An automatic system to control the rate at which green molding sand constituents are added during the mulling process must meet certain basic requirements. The intent of this chapter is to define them. The material that follows is divided into four sections. The first deals with the composition of green sand and the upper and lower limit of each constituent, the second a qualitative description of control systems, the third describes a proposed green sand control system, and fourth a summary.

A. Composition of Green Sand

Green sand, as used by most foundries, is a mixture

of four ingredients. A green sand contains silica sand grains (the principal ingredient), clay, water, and sea-The composition of green sand varies, to some degree, from foundry to foundry, because of differing molding equipment and product lines. Yearley (4) has said that "the foundryman must formulate his sand to suit the molding machine squeeze pressure required to give dimensional accuracy, or he must adjust the squeeze pressure so that it will work with the sand available." The author has observed the latter part of this statement being practiced at General Motors, St. Catharines hodular iron casting facility where automotive crankshafts, flywheels, disc brake calipers, steering knuckles, and exhaust manifolds are produced. green sand used to make all of these products is of the same composition. However, experience has shown that the molding machine squeeze pressure must be increased when making the heavier parts, such as crankshafts and flywheels, in order to maintain dimensional accuracy.

Even with this capability of adjusting the molding machine squeeze pressure it has been found that the composition of green sand must be maintained within close limits in order to maintain product quality. The actual composition and the limits are arrived at through experience and through trial and error. The information on table 3-1 has been compiled from the hourly tests performed on green sand over a one

Table 3-1 GREEN SAND COMPOSITION

This information is from five different sand preparation systems. The composition differs for each system because the sand is used for a different group of products.

CONSTITUENT		WATER (weight %)	CLAY (weight %)	SEACOAL (weight %)
A LINE	low	2.8	9.0	3.8
	target	3.0	9.4	4.2
	high	3.3	9.8	4.4
B LINE	low	2.6	8.8	3.9
	target	3.0	9.4	4.4
	high	3.3	10.0	4.8
C LINE	low	2.5	9.0	4.3
	target	2.8	9.5	4.6
	high	3,1	10,0	4.8
D LINE	low	2.4	8.7	4.7
	target	2.7	9.4	` 5.0
	high	3.1	9.9	5.4
E LINE	low	2.7	9.0	4.7
	target	3.0	9.6	5.0
	h (gh	3.3	10.2	¹² 5.3

(this information is courtesy of General Motors of Canada Ltd. - ST. Catharines Foundry Division)

month period of time at the General Motors Foundry in St. Catharines. The table shows the low and high value of the water, clay, and seacoal constituents measured during the period of time. Also shown on the table are the target values of each sand constituent. It is worthy of note that the high and low values are not the composition limits. As the composition deviate's from the target values and approaches either the high or the low values, casting quality is not maintained. Experience at the aforementioned foundry has shown that it is desireable to maintain the water, clay and seacoal content within 0.10, 0.25 and 0.15 percent (by weight) respectively, of the target values, in order to minimize quality control problems. A composition control system must have the capability to maintain the green sand composition within these limits. Examination of table 3-1 shows that the constituent target values differ slightly for the different sand systems. Typical target values which are used throughout the rest of the report are 2.8, 9.5, and 5.0 percent (by weight) respectively for the water, clay and seacoal constituents. The clay constituent, as mentioned in the second chapter, is a combination of southern and western bentonite. The control system must function so that the clay constituent is 30 percent (by weight) western bentonite and 70 percent (by weight) southern bentonite.

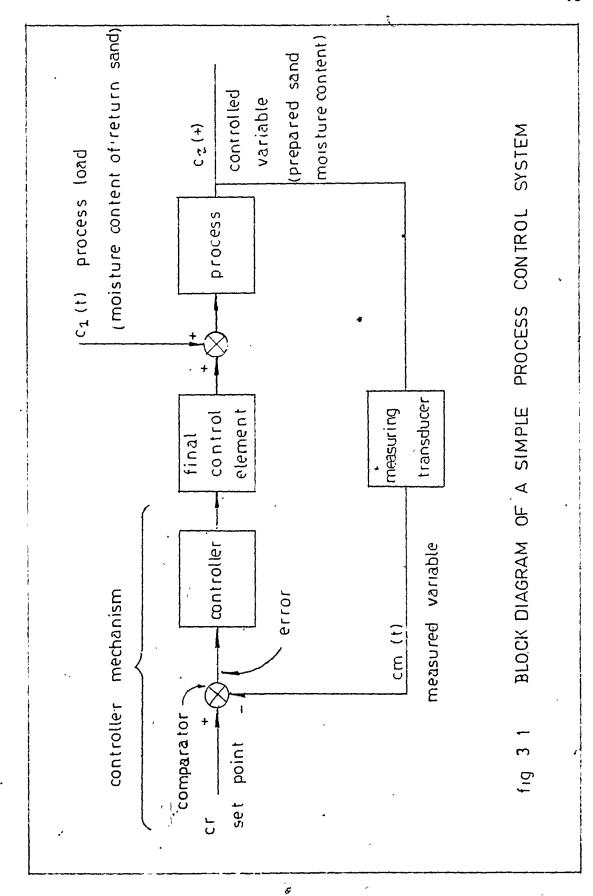
The target values and composition limits are the

basic criteria for the composition control system which is discussed in a qualitative fashion in the next section.

B. The Composition Control System

According to Coughanowr and Koppel (12) a simple process control system may be considered to have four basic components. These are the actual process, the transducer, the controller, and the final control element. A good overall picture of the interaction among these components and the variables involved can be obtained by preparing a block diagram as shown in figure 3-1. Some of the terminology associated with process control is discussed below in terms of this figure.

If, for example, the block diagram of figure 3-1, were a representation of a sand moisture control system, then the process would consist of all the collective functions performed in and by the muller, loading conveyor, water supply line, etc. The process equipment does not include any of the control equipment. There are a number of process variables that deserve to be mentioned. First there is the controlled variable. The controlled variable is what the process control system regulates. The objective of regulation is to maintain the controlled variable at a desired value. Second there is the process load. Load refers to a change in any variable which may cause the controlled variable of the process to change. In a moisture



control system a process load is the moisture content of the return sand. Another is the return sand temperature. High return sand temperatures (above 75°C) result in significant evaporation losses when the tempering water is added. Unless an adjustment is made to the amount of tempering water added when the process load changes, the system will go out of control.

A third process variable, called the manipulated variable, leads to discussion concerning the second basic system component, namely the transducer. The manipulated variable is a quantitative measurement of a physical property that can be correlated to the controlled variable. It would be ideal if the relationship between the controlled and the manipulated variables were linear and independent of any other variables. This, however, may not always be the case. The transducer is a measuring device which changes the units between input and output signals. It measures the manipulated variable and puts out a signal called the measured variable.

These terms can be illustrated by an example. The American Foundryman's Society (5) has proposed that a transducer which measures the capacitance of a parallel plate capacitor with green sand between the plates, be built to measure moisture content. Such a device would be built upon the fact that the capacitance of a parallel plate

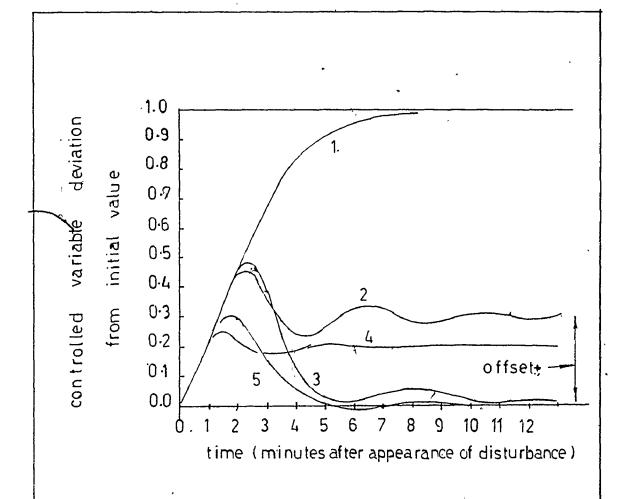
capacitor is given by $C = (A/d)E_O E_F$, where A is the plate area at a spacing of d, Eo is the dielectric constant of a vacuum ($E_C = 8.854 \times 10^{-12} \text{ farad m}^{-1}$), and E_C is the relative dielectric constant of the material between the plates (defined as the ratio of the dielectric constant of the material to the dielectric constant of a vacuum $E_{\Gamma} = E/E_{\Gamma}$). At room temperature water has a relative dielectric constant of approximately 81, while the relative dielectric constants of the other constituents are under 3. Fluctuations in the moisture content affect the capacitance to a far greater degree than fluctuations in any of the other constituents. Provided that the density and temperature remain constant, the moisture content and the transducer capacitance will be linearly related to each In this instance the parallel plate capacitor is a pick-up device. The capacitance of the pickup is the process manipulated variable and also the input to the transducer. The transducer is a capacitance measuring device which has an output known as the measured variable. The units of the measured variable could be electrical current or voltage, the displacement of a needle, or air pressure. The measured variable is transmitted to the third basic control system component, which is the controller.

The measured variable or measured value of the controlled variable is returned or "fed back" to a device

called the comparator. In the comparator, the controlled variable is compared with the desired value or "set point". If there is any difference between the measured variable and the set point an error is generated. The error enters a controller which in turn adjusts the fourth basic control system component, the final control elements in order to return the controlled variable to the set point.

The manner in which the controller adjusts the final control element is termed the control mode. A pneumatic controller may produce an output pressure which is proportional to the error received from the comparator. Such a device is called a proportional controller. Proportional controllers are sold as standard items by several manufacturers (eg' the proportional Taylor Fulscope recorder-controller, made by the Taylor Instrument Company). Other standard controllers add an output signal proportional to the integral and/or the derivative of the error to the proportional response. If a process is subjected to a permanent disturbance such as a sudden unit increase in process load, then the controlled variable will also fluctuate. The curves of figure 3-2 show the behaviour of a typical simple control system utilizing different control modes. This diagram has been taken from the text by Coughanowr and Koppel (12).

In terms of a sand muller without any control being subjected to a sudden increase in return sand moisture



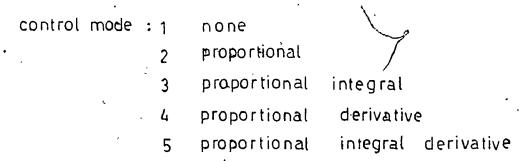
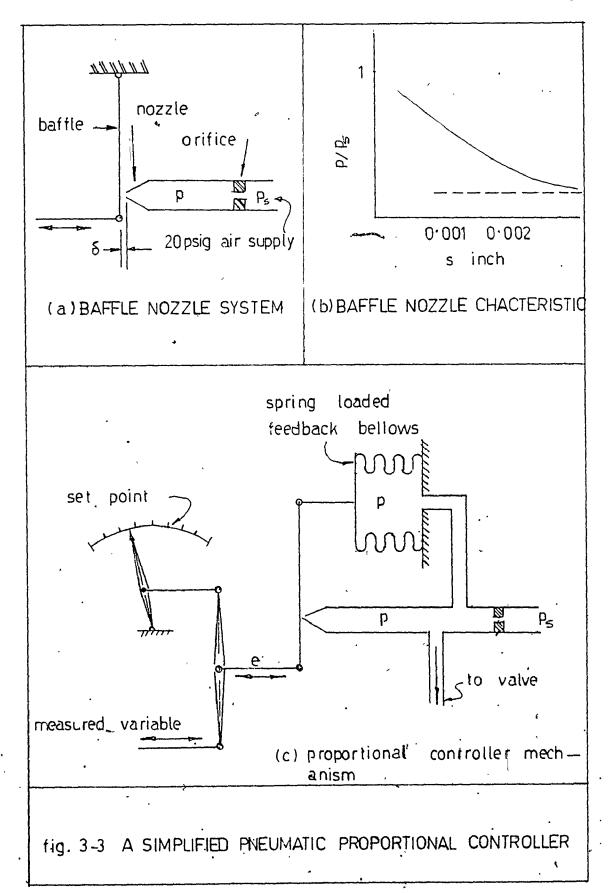


fig. 3 2 RESPONSE OF A TYPICAL CONTROL SYSTEM TO A UNIT STEP CHANGE IN PROCESS LOAD SHOWING THE EFFECT OF VARIOUS MODES OF CONTROL

content, the controlled variable is seen to rise until a new steady state value is reached. The new steady state value is one unit higher than the desired value. With automatic control action is taken to return the controlled variable to the set point soon after the disturbance occurs. With proportional control only the control system is able to arrest the rise of the controlled variable and bring it to rest at a new steady state value. But there is a difference between this new steady state value and the set point which is known as offset. Addition of derivative response reduces the offset and the oscillation of the controlled variable. With proportional integral control the offset is reduced to zero after some oscillation. If the oscillation must be eliminated the proportional integral-derivative mode can be utilized. The addition of each mode means more initial expense and more difficult controller adjustment.

It is desirable to have an appreciation of the actual physical mechanism which receives an error signal and puts out a signal proportional to it. The basic element in an industrial pneumatic proportional controller is the baffle-nozzle system shown in figure 3-3a). The nozzle is usually supplied with 20 psig compressed air. The air passes through an orifice and into a chamber where the pressure, P, is governed by the baffle-nozzle characteristics



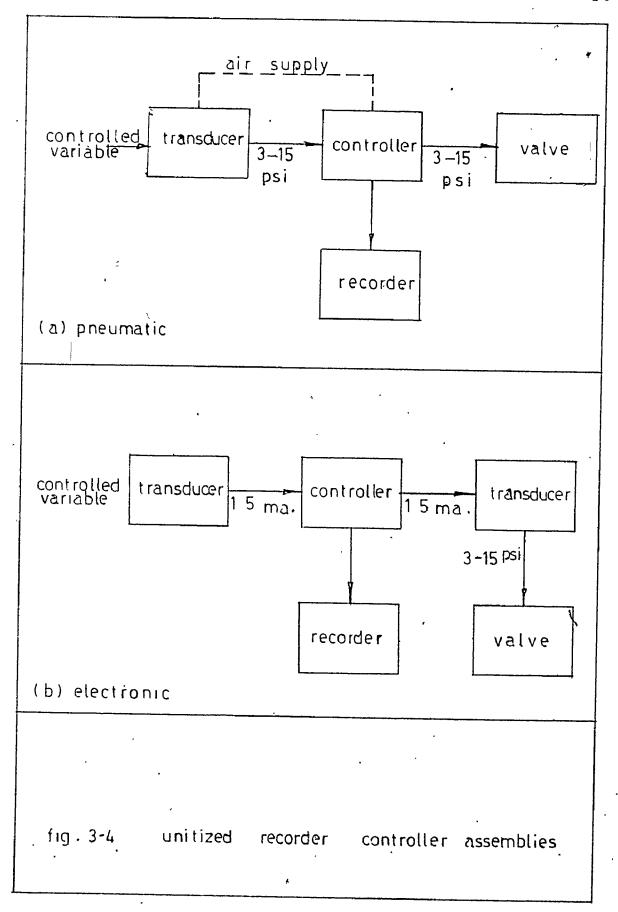
In the past industrial controllers were primarily of the pneumatic type. The practice was to procure a complete control system inside a housing for the measurement of a specific variable. The measuring element was also fastened to the housing on a more or less permanent basis.

A recent trend has been to furnish the measuring element, recorder, controller, and final control element as separate units. This unitized approach offers greater flexibility exemplified by the fact that a temperature pressure transducer of one manufacturer can be used with the controller of another if both use the standard range of

pneumatic signal of 3 to 15 pisg. A unitized pneumatic system for the operation of a control valve is shown in figure 3-4).

Another recent trend has been to replace pneumatic control systems with electronic devices. The new electronic devices are also based on the unitized concept. Some of the advantages, particularly the reliability of the pneumatic devices, have all but been eliminated by advances in electronics technology. Figure 3-4b) is a block diagram of a unitized electronic recorder-controller that operates a pneumatic control valve.

This section about the composition control system is concluded with a few remarks concerning the block diagram of figure 3-1. In addition to providing a good picture of the interaction among system components and variables, the block diagram representation is useful in computing the response of $C_2(t)$, the controlled variable to change in $C_1(t)$, the process load or to changes in $C_1(t)$, the setpoint. Such a computation involves the use of control theory and is outside the scope of this report. A computation of this type would be a valuable tool in the design of the green sand control system once suitable transducers to measure the sand constituents have been identified. Methods of computing the response of a composition control system to load and set point changes are discussed in detail by



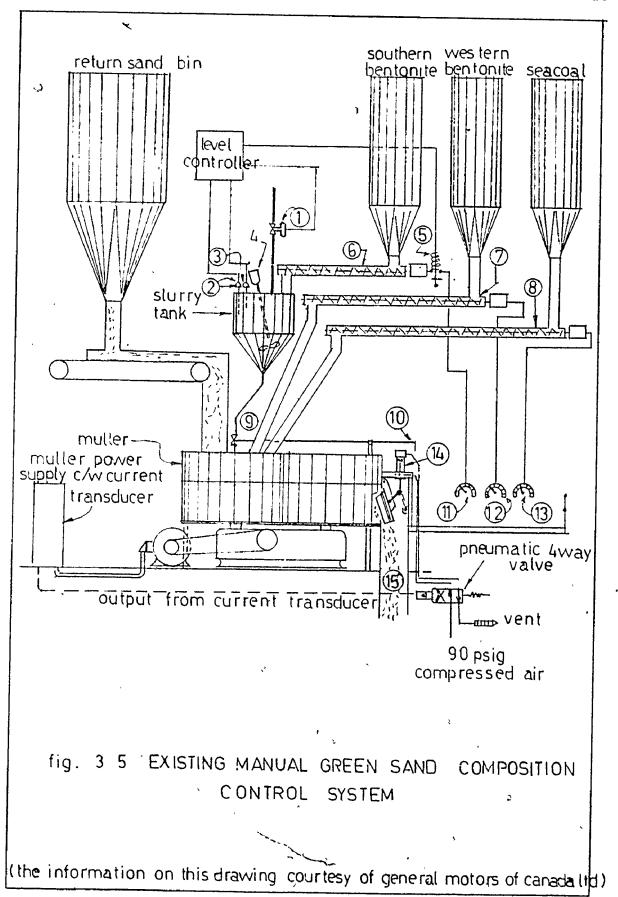
Coughanowr and Koppel (12) and by Considine (15).

C. Specification for the Design of a Green Sand Composition Control System

As stated in the introduction of the report, the ability to monitor the composition of green molding sand and to control the addition of all the constituents would be a significant improvement over the existing manual control methods. A control system capable of performing this function would reduce operating costs and improve product quality.

An examination of the existing manual control system is required before considering its adaptability to automation. A schematic of the manual control system is shown in figure 3-5. It can be seen, from the figure, that automatic controls are used to regulate the amount of sand in the muller and the fluid level in the slurry tank.

The operation of the muller sand level controller, which was mentioned in the second chapter of this report, is based on the fact that the horsepower required (ie the current drawn by the electric motor) to drive it, increases as the amount of sand inside increases. At a predetermined level, the motor current transducer generates a signal which actuates a pneumatic four-way valve. The valve is connected to an air cylinder (item 14) which opens the muller discharge door. In this state the sand is discharged from the muller at a more rapid rate than it is loaded into the muller. As



a result the current drawn by the motor decreases. At a predetermined lower current the signal to the four-way valve is terminated and a spring returns the valve to its original position, which results in the closing of the muller discharge door. During production this cycle is repeated continually.

Water and southern bentonite are mixed together in the slurry tank. The southern bentonite is kept in suspension by an agitator (item 4 in figure 3-5). tank has two conductivity-type level probes. One probe is, a low level indicator (item 2) and the other a high level indicator (item 3). In the event that the slurry level is low (ie neither probe extends into the slurry), the level controller simultaneously generates a pneumatic signal which opens the water valve (item 1) and an electrical signal which operates a relay (item 5), activating the southern bentonite auger conveyor (item 6). of operation, slugry is generated more rapidly than it is used. As a result the slurry level increases. When the slurry level rises to the point where both probes are immersed, the level controller terminates the signals to the auger conveyor and the valve and filling ceases. During production slurry flows into the muller continuously. flow rate is determined by the condition of the return sand and regulated by a manually operated butterfly valve (items

9 and 10). As a result the slurry level decreases. At the low level the filling action is re-initiated. the slurry level fluctuates, the fluid head at the control valve (item 9) also fluctuates. At a particular muller installation at General Motors in St. Catharines, the slurry level was observed to vary by six inches. The liquid head above the control valve varied from 19.5 to 20.0 feet. The flow is governed by the equation $Q = CA\sqrt{2gH}$, where Q is the flow quantity, C is an orifice constant, A is the area of flow, g is the acceleration of gravity, and H is the liquid head in feet. If, for example, the return sand moisture content is 0.8 percent, then 2.0 percent moisture must be added at the muller. If all other process loads are stable, then the controlled variable will fluctuate between 2.79 and 2.81 percent moisture. While it must be considered, the effect of this particular process load is small. It can be neglected.

It is evident from figure 3-5 and the mode of operation that the rate at which southern bentonite is added is governed by the rate at which moisture is added because they are mixed together and by the auger conveyor speed control (items 6 and 11) which enables the operator to change the southern bentonite concentration in the slurry tank. Experience at General Motors - St. Catharines has shown that a dbange in moisture requirement is generally accompanied by a change in

clay and seacoal requirements. Adding one of the clays along with the water does not present a control problem. However there is not a direct relationship between the moisture, clay, and seacoal requirements of return sand. Separate controls for each constituent are required.

When considering automation an important element is simplicity. Examination of figure 3-5 shows that four constituents are added to return sand. A simplification can be made by leaving the slurry system (ie items 1,2,3,4,5,6, and 11 on figure 3-5) intact and applying automatic controls to the slurry, western bentonite, and seacoal addition only.

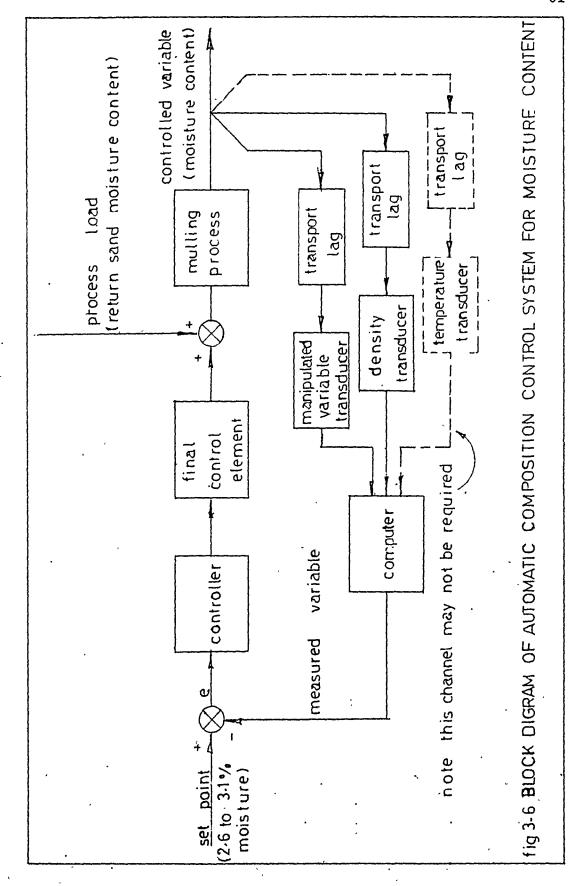
Investigation has shown that it is difficult if not impossible to determine the amounts of different clays in a mixture, even if utilizing laboratory analytical techniques. The author is of the opinion that a clay transducer, when it is developed, will measure the total amount of clay in green sand only. Such a transducer, in a control system, will detect changes in the overall clay requirements. The controller will adjust only the western bentonite auger conveyor to make immediate compensation over a longer term, the western bentonite to southern bentonite ratio will be maintained at the required 30 to 70 level by manually adjusting the southern bentonite auger conveyor speed control (item 11 of figure 3-5) when the

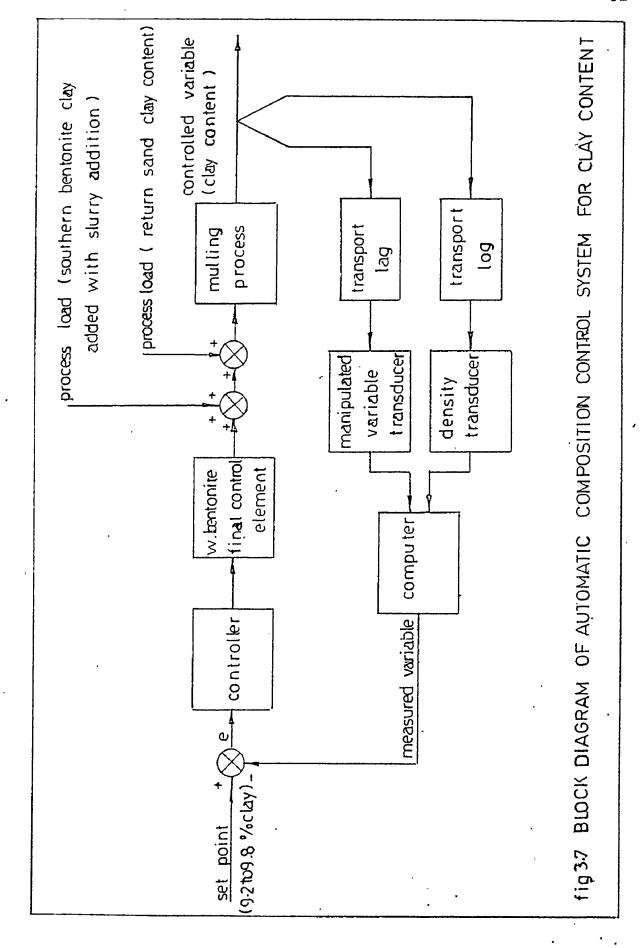
system is periodically calibrated, similar to the way the ratio is controlled under the manual control mode.

As in the manual mode of control, the water and southern bentonite will be added together at a rate determined by the moisture requirement of the return sand.

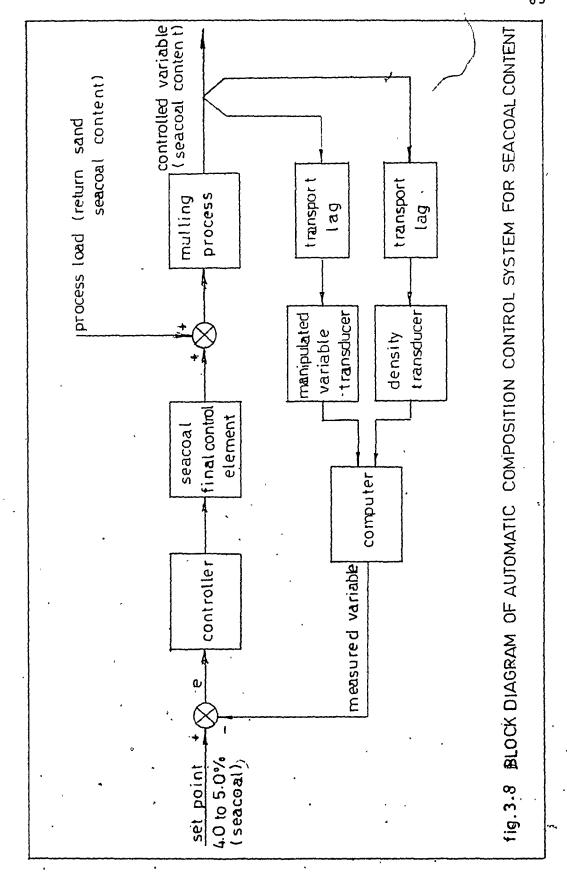
The proposed automatic control system must meet the following specifications.

- 1. Three subsystems to control the water, clay, and seacoal green sand constituents shall be provided. The block diagrams for these subsystems are shown on figures 3-6, 3-7, and 3-8. The blocks labelled transport lag are included because the transducers will be located outside the muller at point 15 on figure 3-5. It will take a certain amount of time for the sand to reach the transducer.
- 2. a) The transducer precision limits shall be a maximum of plus or minus 0.1, 0.25, and 0.15 percent by weight for water, clay, and seacoal respectively. The precision or reproducibility of a transducer is defined as the closeness with which it repeats indications when identical values of the controlled variables are measured. The precision of a transducer is closely related to its accuracy which is the closeness of agreement between the observed result and the known or true value. In this application, precision is more significant than accuracy because measurement is for the purpose of control and not to determine absolute values.





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- b) It would be desirable if the measurement technique were of a non-destructive nature. This matter is discussed in more depth in chapter 4, section B.
- c) Other variables such as density and temperature, which, depending upon the measurement selected, will affect the manipulated variables, must also be measured. Correction for the error introduced by these variables must be introduced.
- d) The minimum range of the transducers measuring the water, clay, and seacoal contents are 2.0 to 3.5, 8.0 to 11.0, and 3.0 to 6.0 percent by weight respectively. Allowance shall be made for the fact that the manipulated variables will be influenced by factors such as density and temperature. The minimum range of the density and temperature transducers shall be 1.0 to 1.5 grams per cubic centimeter and 10°C to 100°C respectively. By definition the range of a transducer is the span of concentrations to which it responds without adjustment.
- 3. a) The controllers for the water, clay, and seacoal constituents shall have set points adjustable over the range 2.6 to 3.1, 9.2 to 9.8, and 4.0 to 5.0 respectively.
- b) The controllers for all three constituents shall operate on a proportional integral or a proportional—integral—derivative control mode. This mode is selected to eliminate the possibility of controlled variable offset.
 - c) The controllers may be pneumatic or electronic.

- d) The controlled variables shall be recorded.
- 4. The slurry final control element shall be an air operated Saunders diaphram valve. The auger conveyor final control elements shall be variable speed drives.

D. Summary

Automatic control systems similar to those described in this chapter have been successfully applied to numerous different production processes. In many instances the basic control systems components can be purchased on an off-the-shelf basis. In the case of green sand all components are readily available except for the transducers. It appears that suitable transducers have not yet been developed. The next chapter is a survey of different analytical measurement techniques that were investigated with the intent of developing transducers capable of measuring green sand constituents.

CHAPTER 4

DETERMINATION OF GREEN SAND COMPOSITION

As mentioned previously, the basic components of a composition control system for green sand are all readily available except for the transducers. The subject matter of this chapter deals with instrumental techniques for the determination of chemical composition variable and the feasibility of adapting one or more of these techniques into a transducer or series of transducers to measure the constituents of green molding sand. The material that follows is divided into three First, the results of a literature survey of methods to determine green sand constituents that have been attempted or suggested are presented. Second, the general physical chemical phenomena and the practical considerations involved in the measurement of chemical composition are discussed. results of the literature survey are given in the final section of this chapter.

A) Literature Survey of Existing Monitoring Practice

Water is the most important constituent of green sand in the sense that a slight variation in its concentration has a greater effect on its properties and performance than any of the other constituents. For these reasons several automatic methods of measuring the water content of green sand have been attempted and several have been suggested in the literature which to the knowledge of the author have never been tried.

The clay and seacoal constituents in green sand are more difficult to measure than the water content. To the knowledge of the author, a method of measuring these constituents, that would also be useful in an automatic composition control system, does not exist in actual foundry practice. Only one method of measuring clay and seacoal on a continuous automatic basis has been suggested in the literature. At General Motors in St. Catharines, for example, clay and seacoal are measured by gravimetric means after systematically removing these constituents from the sample. All existing routine analyses for these two constituents are done by methods involving grab samples and manual labour.

This section is a review of analytical methods that have been developed or suggested for the measurement of moisture, clay, and seacoal content in green sand. Considerable work has been done to control moisture content in other industries. Methods that are used elsewhere are also included in this survey. The response of an analytical instrument to the concentration of a particular

constituent is often affected by the density and temperature of the sample. Methods for the measurement of these variables have also been included in this review.

- Drying and Weighing: The standard method of obtaining the water content of green sand is to obtain a 50 gram sample, oven dry it to a constant weight at a temperature between 105 and 110 C and then reweigh it. The difference in weight multiplied by a factor of two is the water content in weight percent. For accurate results the drying process takes at least one hour. This technique is used as a standard to calibrate other methods. According to Considine (15) several automated devices which utilize this concept are available. Use of such a device eliminates much of the manual labour involved and reduces the amount of time required to make a measurement. With the use of one such device, the Speedy Moisture Teller, manufactured by the Harry Dietert Company Ltd. (16) made specifically for measuring the water content in green sand it is possible to obtain a result in about two minutes. The drying time is reduced by forcing heated air through the sample. Even so the device is still periodic in nature and as such not practical for an automatic control system.
- 2) Gas Bomb Technique. The water content can be estimated rapidly by measuring the volume of acetylene gas generated when a weighed sample of sand is mixed with an excess of calcium carbide powder in a closed container. This method is also periodic in nature and as a result does not lend itself to an automatic control system.
- 3) Electrical Conductivity. The fact that the electrical conductivity

of green sand increases as the water content increases has been reported by the American Foundrymans Society (5), by the British Cast Iron Research Association (17), and by Chunaev (10). Both the AFS and the BCIRA report that the packing density of green sand has a more pronounced effect on electrical conductivity than the moisture content. They also report that the conductivity is affected by the clay content, seacoal content, coke content, and temperature. All of these variables would have to be measured before this method could be used. It has also been reported by Considine (14) that conductivity meters measure only the path of least resistance in a granular material. This inherent weakness would tend to give inaccurate results.

At least one moisture control device, that measures the electrical conductivity of green sand, has been developed specifically for the foundry industry. This device namely the Hartley Series XVII Controller (19), is commercially available at the present time. The device has the capability of compensating for changes in conductivity due to temperature fluctuations. The manufacturer (19) of the Hartley Controller specifies that their device will work properly only if the return sand additives are "fairly consistent" at all times. In the opinion of the writer, automatic controls would not be required if this condition were met. In addition to this the manufacturer has stated that the precision of this particular instrument is plus or minus 0.25 percent water. This is typical of electrical conductivity moisture measurement

instruments. They are not precise enough to be used in this particular application.

- 4) Humidity Sensing. Considine (14) has reported that the water content of paper webs and other sheet materials has been controlled with the use of humidity sensitive primary elements, which are placed just above the webs. The report indicates that this type of a system works well for this application. The accuracies obtained (plus or minus two percent water content for paper webs) are not good enough for use on molding sand. The abrasive nature of molding sand would dictate that a humidity sensitive element be placed in the air somewhere where the sand could not come into contact with it. An equilibrium between the humidity of the air and the sand moisture would have to exist in order that the measured variable be representative of the sand water content. This equilibrium does not exist because the air in the sand handling equipment is exhausted to control dust. Humidity sensing is not suitable for measuring sand moisture content.
- Capacitance. A capacitor is formed when two or more electrically conductive elements are separated by a less conductive medium called a dielectric. If the shape, size, and spacing between the conductive elements remains fixed, the capacitance of such a device varies directly with the dielectric constant of the intervening material.

 If moulding sand is the intervening material, then changes in the

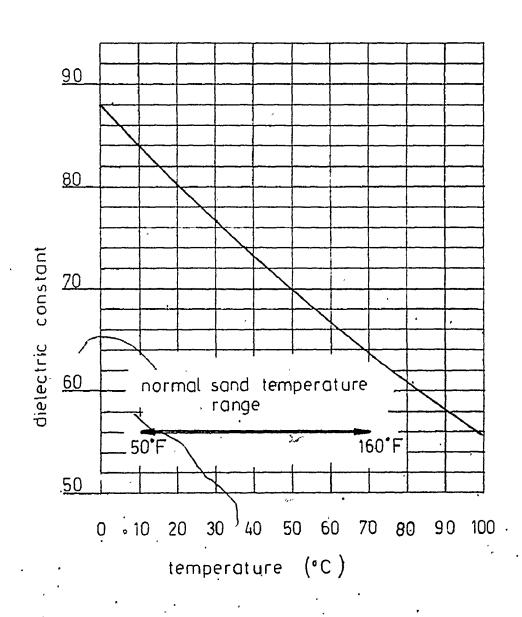
water content can be estimated from changes in the capacitance. This type of an element works well provided that the packing density of the sand remains constant, the conductivity is stable, and provisions are made, to adjust the output according to changes in the temperature of the sand (See fig. 4-1). No significant change in capacitance arises due to normal changes in additions other than water became the dielectric constant of water is high compared to other constituents. Figure 4-1 shows that the dielectric constant of water at 20°C is about 80 while the other constituents have dielectric constants of about 3. The data used to construct figure 4-1 was obtained from reference (20)

This primary element is sensitive to the packing density because the amount of water in the intervening space can be increased if the packing density is increased. The difficulty with density is compounded by the fact that molding sand swells as the water content increases. Thus for uncompacted and partially compacted molding sand, any tendency to increase a capacitance reading due to an increase in the water content is at least partially nullified by the lower density. For example, according to figure 2-6, one cubic foot of riddled sand at 3.0 and 3.6 weight percent water weigh 66 and 50 pounds respectively. The actual weight of water in these two cubic feet of sand is 1.98 and 1.80 pounds. A sample of the drier sand actually contains more water. It is essential that density be measured.

The British Cast Iron Research Association (17) has done

Fig. 4-1. <u>DIELECTRIC CONSTANT OF WATER</u>

<u>VERSUS TEMPERATURE</u>



work to determine whether capacitance measurement would accurately indicate the moisture content of green sand. device was designed, built, and eventually tried out in an actual foundry. The effect of density fluctuations were reduced by compacting the sample to the maximum bulk density which required compaction pressures in excess of 300 psi. Figure 2-6 shows that the change in density of the compacted material with change of water content is small when compared with the behaviour of the material in its riddled condition. Proving trials showed that temperature variations in the sand affected the repeatability of the instrument. The investigatwere of the opinion that the error introduced by temperature variation could be simply corrected. It was also reported that a high coke content prevents a satisfactory measurement of water content by capacitance measurements. is formed by the action of the molten iron upon the seacoal additive. Normally the coke content of a used sand is about two percent. When the coke content approaches four percent, the sand becomes highly conductive and no longer acts as an effective dielectric. The investigators concluded that sands with a high coke content could not be tested by this method, and that a repeatability of the order of plus or minus 0.5 percent moisture can be achieved on a synthetic sand. This is not adequate for this application.

- 6) <u>Multiparameter</u>. The multiparameter method is reported to be capable of monitoring the water and clay content of molding sand and of making compensation for changes in the packing density of the sand. This method was evaluated experimentally (see Chapter 5) for a number of reasons.
 - a) Only the principle of operation was described.
 - b) The reported capability of the method made it very attractive .
 - c) The equipment required to implement this method was readily available.
- 7) Neutron Scattering. The use of high energy neutrons to measure the water content of materials has been described by Wormald and Britch (21), Stalhuth (22), Waters and Moss (23), van Bavell (24), and others.

High energy neutrons which are emitted from a radioactive material such as radium-beryllium, engage in elastic
collisions with the atomic nuclei of all elements. Because
they are almost equal in weight, a high energy neutron loses
much more kinetic energy in a collision with a hydrogen atomic
nucleus than with the atomic nucleus of any other element. The
process whereby high energy or "fast" neutrons lose kinetic
energy as they collide with hydrogen atomic nuclei elastically 's
is called thermalization. A beam of fast neutrons directed at
a material containing hydrogen is partially thermalized resulting in a flux of slow speed neutrons which emanates from the

material. The intensity of the flux is directly proportional to the amount of hydrogen in the material. The intensity of this flux canbe measured with an appropriate counter. In materials where hydrogen is the main element of low atomic weight and is largely contained in free water, the thermalized or slow neutron count is a direct measure of water content.

Primary elements, for moisture control systems, have been constructed utilizing the principle described above. An advantage is independence of sample temperature. Disadvantages cited in the reference material are that 1) up to two minutes is required to obtain a stable slow neutron count, 2) the slow neutron count is dependent upon the sample packing density, and 3) the slow neutron count is affected by hydrogen in other sample constituents.

For molding sand, independence of sample temperature is a definite advantage. The sand temperature may vary between 50°F and 180°F, which is significant for some of the other measurement techniques. Dependence upon sample packing density does not present any significant problem. Several neutron moisture measurement systems (25, 26) are commercially available with a gamma ray bulk density measurement package. In this system the slow neutron count is corrected for variations in density. The fact that the slow neutron count is affected by hydrogen in other sample constituents does present a significant problem. The total amount of hydrogen in the seacoal is about the same

as in the moisture. One hundred pounds of green sand with a seacoal and moisture content of 5.0 and 2.8 percent respectively contains approximately 0.56 lbs of hydrogen. Seacoal accounts for 0.27 lbs and moisture for 0.29 lbs. Seacoal would have to be measured by some other means if the neutron method were used for moisture, because both constituents fluctuate significantly The measurement of seacoal is discussed in a subsequent section of this chapter.

The reference material indicates that the best accuracy obtainable, with this type of a moisture measurement system, is plus or minus 0.25 weight percent water. This in itself rules out the method because the accuracy requirements are not met.

8) Nuclear Magnetic Resonance. The use of nuclear magnetic resonance as an instrumental technique for measuring moisture in materials has been described by Ewing (27) Rollwitz (28) and others. Nuclear magnetic resonance is the name given to a phenomenon whereby two mutually perpendicular magnetic fields, the one stationary and the other varying at a radio frequency, interact with materials having atomic nuclei that spin. To have a nuclear spin the nucleus must have an odd number of neutrons or protons such as 1 H, 19 F, or 29 Si. The effect of the stationary magnetic field upon a material containing water is to cause the spinning hydrogen nuclei to precess about the direction of the field. Ewing (27) reports that the spinning

hydrogen nucleus can orient itself with the stationary field in either of two ways, which means that it can occupy two energy levels. The application of the alternating field at the precessional frequency, f to the sample, results in the absorption of energy by the hydrogen nuclei in the lower energy state. The absorption can be observed at the output of a detector.

Unlike neutron absorption, NMR can differentiate between hydrogen that exists in different chemical environments. For example the hydrogen in water would absorb at a different combination of stationary magnetic field strength and f, the frequency of the alternating field, than the hydrogen in seacoal. The amount of energy absorbed at each particular combination would be a measurement of the amount of water and seacoal present in the sample. Galbraith, Ladner, and Stacey (29) have investigated the possibility of using this method to measure the moisture content of coals and coke. They report that moisture can be measured with an accuracy of †1 percent of the total moisture content up to 30 percent moisture. This means that at 3 percent moisture it is possible to make a measurement accurate to within † 0.03 percent water. This clearly exceeds the accuracy requirements for green molding sand.

There are, however, a number of other difficulties with this method. a) The stationary magnetic field into which the material under test is placed must be very powerful and very uniform. As a result the maximum sample size normally used is about one inch in diameter. Feeding an

abrasive and cohesive material through an opening of this size on a continuous basis presents a difficult problem.

- b) The measurement would have to be corrected for the packing density of the sand. Obtaining the density of the sand in the sampling zone is also difficult.
- c) The cost of this type of an instrument is high.
- The presence of ferro magnetic materials in the sample d) causes problems with the instrument. According to Gray (30) ferromagnetic constituents detune a high resolution There is a significant amount of iron in instrument. green sand. First, there are iron particles which evade the magnetic separators in the sand system. Second, used sand grains become coated with a layer of cast iron. The author found that virtually all of the sand grains in a random handful from any of the sand systems at General Motors could actually be picked up with a small permanent magnet. The manufacturer of an NMR process moisture analyzer (31) stated that there would not be any response from their instrument with a sample of sand in this condition.

A moisture analyzer using the nuclear magnetic resonance principle is not suitable for this particular application.

9) Microwave Absorption. The absorption of microwave radiation has been used to measure the moisture content of a number of materials. Lindberg and Ternstrom (32) describe the application of the microwave absorption technique to the measurement of moisture in pulp and paper products. Taylor (33) describes a microwave absorption method of measuring the watercontent of fertilizer and soap powder.

When microwaves pass through a material, their radiant power is reduced because of absorbtion. For many materials the amount absorbed is primarily dependent upon its water content the power absorbed in the dry state being almost negligible. Water content can thus be estimated by measuring the microwave power absorption of the material. This equipment is sensitive to the amount of water per unit volume, hence, for a granular material, like molding sand, the power absorbed is sensitive to the packing density. An advantage of the method is that it uses a relatively large sample which means that it is more likely to be representative of the material from which it is taken than some of the other instrumental methods.

Godding and Bird (17) have proposed that microwave absorption be used to measure the moisture content of green molding, sand. These two investigators developed a prototype unit in which the difficulty of the packing density was solved by using a mechanical device that compacted a slug of sand inside

a steel tube. It was found that sand, regardless of its composition, could be compacted to about the same density by a pressure in excess of 300 psi. The steel compaction tube had a pair of opposed windows for the microwave transmitter and receiver. These windows were closed with plastic inserts which wore out rapidly and required replacement about once every 16 The slug of sand produced was tested for water content. The repeatability was reported to be plus or minus 0.3 percent moisture The normal variations of active clay, dead clay, seacoal, and coke constituents, which affect the absorption of microwave power, contributed to the measurement error. The author does not know of a way of correcting for the variation in these constituents. The investigators noted that normal variation of sand temperature introduced a significant error. They concluded that in an industrial setting, automatic temperature compensation would have to be incorporated into the unit.

Unfortunately, the repeatability of the method dictates that it does not lend itself to the measurement of moisture in green sand.

10) Infrared Absorption. The near infrared spectrum contains two radiation bands, centered approximately on wavelengths of 1.40 and 1.94 microns, which can excite water molecules by a reasonant absorption phenomenon. If infrared radiation from either of these bands is incident upon water, then the vibrational state of the O-H bond will be excited to a hich.

energy level. In the process of exciting the water, some of The amount by which the the infrared radiation is absorbed. intensity of a beam of resonant infrared tradiation is diminished, is proportional to the amount of water in its path. A similar phenomenon occurs with other infrared radiation bands for many Numerous laboratory and process analysis instruments compounds. have been constructed on this principle. A requirement of all laboratory instruments based on the absorption of infrared radiation is that the incident radiation be transmitted through the sample. As a result the sample must be a transparent solid, clear liquid, or a gas. Sand, because it is an opaque solid, cannot be analyzed with a conventional laboratory infra red instrument. The use of these conventional instruments has been described by Ewing (27), by Willard, Merritt, and Dean (34), and by many others.

A recent development in the area of infrared absorbtion is a specialty device for the measurement of the moisture content of materials. The incident radiation is reflected from the material. The design of this type of an instrument has been described by Considine (15) and by the Anacon Instrument Company (35). This type of instrument has a tungsten filament infrared source and two filters mounted on a spinning disc called a chopper. One filter passes an absorption band of radiation centered on the 1.94 micron wave length, while the other passes a

nonabsorptive band centered on the 1.80 micron wavelength. non-absorptive band, also known as the reference band, is not affected by water or any other constituent of the material to The chopper also isolates the two bands sequentbe monitored. ially. After it leaves the chopper the radiation is directed at the material. Some of both the absorption and the reference band is reflected back at the instrument, where a mirror directs it at a lead sulphide photoconductive detector. The chopper and the detector are synchronized so that both the absorption and the reference bands are converted into separate electrical signals. The reflectance of any material is dependent upon three factors; its surface absorption, surface scatter, and special absorption properties due to chemical composition. The surface absorption and scatter properties diminish the intensity of the reflected radiation from either band in exactly the same The water content of the material diminishes only the absorption band. The reference band is used to remove the effects of surface absorption and scatter from the absorption band. The net effect is an electrical signal proportional to the amount of water on the surface of the material. With the Anacon instrument, users have the option of selecting an electrical voltage or current output signal. Because the unit is sensitive to the amount of water present; fluctuations in the packing density would introduce error. In applications where the packing density is uniform, the manufacturer advertised an accuracy of better than plus or

minus 0.1 percent.

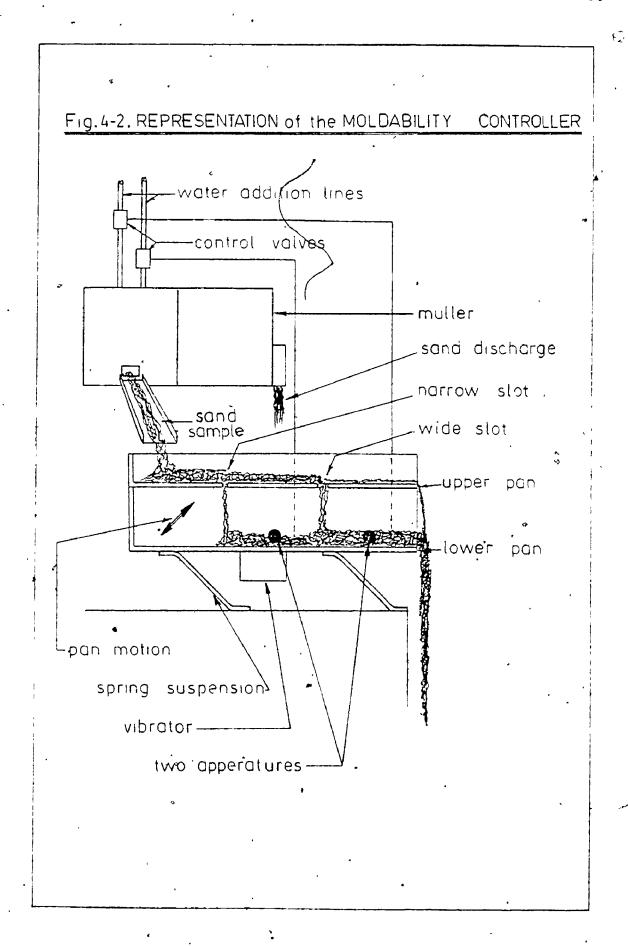
The manufacturer was contacted concerning the instrument, because the reported accuracy was better than actually required. A test was set up. It was found that there was very little response from the instrument even for moisture content in excess of (10) percent. The problem appeared to be that the clay absorbed the water and shielded it from the absorption band. The sales representative made inquiries at their head office and discovered that the instrument had been tried out on molding sand at a Ford Motor Company Foundry, again with negative results. This method does not appear to be suitable for this application. Further investigation is required to discover the exact reason for the failure of the device.

Moldability Controller. Molding sand must possess certain mechanical properties in order to be used to produce good castings. These include moldability, flowability, green compression strength, hot strength, and permeability. The desired mechanical properties are obtained if the correct amount of each constituent is present in the sand. Conversely, if a sand mixture has the desired mechanical properties, then the correct amount of each constituent is present. The moldability controller, developed by the Harry W. Dietert Company (16), utilizes this principle. This device has been described in one of the leading foundry trade jour-The mechanical property utilized by the controller is the moldability of the sand. Moldability appears to be related to the tackiness of molding sand. It is measured by the ability of a mass of riddled or loosely packed sand to bridge a slot in a The width of the slot that vibrating pan .

a sand is able to bridge is a measure of its moldability. The instrument is illustrated in figure 4-2. Each of the two aperatures in the sides of the lower pan allow light to illuminate a photocell. Each photocell controls a valve in a separate water addition line. If the sand bridges both gaps in the upper pan, then the valves in both lines are closed. If either apperature is blocked by a buildup of sand on the lower pan, then the water valve in the corresponding line is opened. Control of water content is achieved as described below.

- a) The first slot is set at the lower allowable moldability and the second at the upper limit.
- b) A small quantity of sand is fed from the mixer bnto the pan as shown in figure 4-2.
- c) If the sand is dry, its moldabilty will be very low and it will fall to the lower pan. Both water lines to the mixer will be opened.
- d) Both lines remain open until the sand develops sufficient moldability to bridge the first (narrow) slot. Once the first slot is bridged the first appearature is no longer blocked and the first water line is closed.
- e) Depending upon the water requirement of the sand, the moldability could continue to increase until the second slot is also bridged. The second water line would then also be closed.

The moldability controller allows the moldability of the sand to



drift between the upper and lower moldability limits.

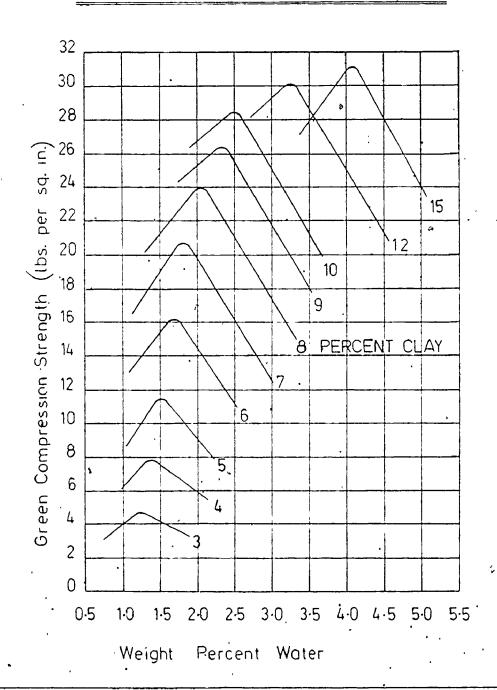
The following is an evaluation of the moldability controller.

- This instrument does not control the water or clay content. The same moldability can be achieved with different claywater combinations. There is no published data relating water and clay content to moldability as measured by the moldability controller. However figure 4-3 shows that the same green strength, which like moldability, is related to the tackiness or bonding ability of molding sand, can be achieved with many different clay-water combinations. The manufacturer (37) indicated that a similar relationship exists for moldability, making 1t necessary to use conventional control procedures, including the hand squeeze method, along with the automatic moldability control in order to maintain the clay content at the required level. If this is done then the moldability controller will maintain the water content at the required level. The clay content is easier to maintain at a constant level than moisture, because it is a non-volatile constituent.
- b) The experience of foundries has been that these controllers cannot replace the muller operator. The controller assists the operator by improving the response to variations in the return sand moisture content.
- c) The instrument is simple, rugged, and inexpensive. A muller installation can be equipped with one of these units for approximately \$10,000.00.

Fig 4-3. GREEN STRENGTH VERSUS WATER

CONTENT FOR MOLDING SAND BONDED WITH

VARYING AMOUNTS OF WESTERN BENTONITE



- d) The author is of the opinion that every foundry should seriously consider the use of moldability control units for its sand systems, based on the potential for improvement to product quality. In many situations the acquisition of these control units will, in fact, realize lower operating costs through a reduction in the casting scrap rate. However, this is not a complete solution to the control problems because the conventional sand control procedures are still required. A system capable of measuring and controlling the chemical composition would be superior because it would enable a substantial labour reduction.
- 12) Clay and Seacoal Measurement Methods. A number of laboratory methods are used to measure the clay and seacoal constituents in green sand. These methods were mentioned in the second chapter of this report.

The method of elutriation for the measurement of clay content, adopted as a standard by the American Foundrymans Society (38), involves washing all fine material, less than 20 microns in size from the sample and weighing the residue. The percent weight loss is the AFS clay content. The nickel sulphate test for active clay content (10) depends upon the adsorption of nickel ions by active clay in a nickel sulphate solution. The remaining nickel is formed into a colored complex by adding dimethyl glyoxime. The amount of active clay is determined by

filtering the solution and measuring the intensity of electromagnetic radiation (540 nm) transmitted through the sample on
a spectrophotometer. The methylene blue test (10) for clay
involve the titration of a sample of sand with a methylene blue
solution. The amount of solution required to reach an end point
is a measurement of the active clay content. The nickel sulphate
and methylene blue tests are described in more detail in Appendix III.

A new method based upon the compactability of a green sand mixture has been suggested (39) as a way of measuring the clay content. Compactability is determined by riddling a sand sample into a standard 2 inch diameter by 2 inch high specimen The tube is filled to overflowing and the sand is gently struck level with the tube top. The sand is then compacted with three blows of the AFS standard 14 lb. rammer. The decrease in height is divided by the initial height to obtain a figure that is expressed as a percentage compactability. The compactability has the following implications. The minimum density point of thoroughly mulled green sand exists at approximately 60 percent compactability. This can be seen from figure 2-6. As the moisture content decreases from the minimum density point, the thickness of the water-clay coatings around the sand grains decreases with the result that more sand can be initially riddled into the specimen test tube. The compactability is therefore decreased. In conjuction with moisture determinations, compactability.

Seacoal is determined by measuring its volatile constituent A sample of sand is placed in an oven at 1800°F and held at that temperature until its weight is constant. The weight loss is related to the seacoal content (38).

The measurements for clay content by elutriation, the methylene blue test, and the nickel sulphide test are of a periodic nature and as such are impractical for automatic control systems. Provided that a method of measuring moisture to adequate accuracy could be found the method of compactability does hold potential for use with an automatic control system. This is elaborated upon in a subsequent section of this chapter. The measurement for seacoal is also of a periodic nature and as such is impractical for use with an automatic control system.

technique is often affected by the temperature of the material under analysis. There are several methods of measuring temperature that are compatible with automatic control systems. According to Considine (15) resistance temperature detector elements are available with a repeatability of better than plus orminus .10 F. Thermocouple and thermistor systems could also be used but with less advantageous repeatability characteristics. When the temperature is a significant factor in a measurements technique, a

resistance temperature detector (RTD) element could be used to compensate for the error.

Density Measurement. The response of a measurement instrument is proportional to the amount of the desired constituent in the sample, which is determined by its concentration and the density of the sample. When an on-line, continuous, analytical instrument is used to measure a constituent of green sand, the density must also be measured. The method of measuring the density of bulk materials, most often encountered in the technical literature, is based on the Compton scattering of medium energy The essential components of such a system are gamma radiation. a radioisotope source in a lead-shielded holder and a detector fixed on either side of the sample. Density is determined by measuring the attenuation of gamma radiation. Accuracies as good as plus or minus 1 percent have been reported for soils (21) and as good as plus or minus 0.03 percent for slurries moving through pipelines (15). The accuracy exceeds the requirements for the measurement of the constituents of green sand. The method can be incorporated into an automatic control system.

B) General Physical-Chemical Phenomena of Chemical Composition . Measurement

A suitable monitoring method that could be incorporated into an automatic control system does not exist in actual foundry practice for any of the green sand constituents. In view of this fact the instrumental techniques of analytical chemistry were reviewed for the feasibility of using one or several of these techniques for green sand constituent measurement transducers.

One of the problems that confronts a person seeking to find a method of determining a particular chemical composition in a process stream is that of selecting the best possible analytical technique and/or instrument from the large number that have been developed. In response to this problem, the methods of analytical chemistry have been classified for the purpose of providing a systematic approach to the selection of a technique for a given analysis problem by several individuals. Ewing (27) has approached the problem from the point of view of laboratory analysis, ie. the instrument or technique is to be used by a technician as a tool for the analysis of a sample about which little of perhaps no advance knowledge in regards to its composition is available. Considine (15) has approached the problem from the point of view of determining and controlling a chemical composition in a process stream. In a control application the instrument operates unattended, analyzing a sample about which

considerable advance knowledge is known in regards to what kinds of material will be present and their expected ranges of concentration. The approach by Considine is more applicable to the selection of a technique for measuring the constituents of green sand. It is discussed below.

Consdine (14) has devised a useful reference, framework which enables a more systematic approach to the selection of an analysis method. Table 4-1 which is reproduced from Considine (14), separates the analytical methods into four categories based on the fact that chemical composition is determined by subjecting a material to an external source of energy and measuring the consequences of the interaction between the material and the energy source. The analytical methods are separated into four groups because the external energy source may be in one of four fundamentally different categories, which are:

- 1. Electromagnetic radiation
- 2. Chemical Affinity or Reactivity
- 3. Electric or Magnetic Fields
- 4. Thermal of Mechanical Energy.

An understanding of these interactions between matter and an external energy source can be built upon a knowledge of the structure of atoms and molecules. All known matters made up of a complex but systematic arrangement of particles having mass and electric charge. For all practical purposes these particles are neutrons which possess mass but no charge, protons which possess mass and a unit positive charge, and electrons which have

Table 4-1 Generalized Relationships between Matter and Energy That Can Be Measured to Ascertain Chemical Composition

	•	•
interaction with thermal or west hand tringsy Measurement of the results of apply- ung the ring or need wifein tarry to a system in terms of energy transmission, work done, or changes in physical searce	The thermody name relationship involving the physical state and stand-correct of any substance permits analysis and identification of mixtures of soliding de determination of freeing or boiling points and upon the quantification measurement of physically separated fractions. Useful infortural-conductivity and visconity mation erg factors derived from the rand-conductivity and visconity measurements.	CIG EX. Trylectively
Reaction to electrical and myrineth filler. Measurement of the current, i.oli. The or flix changes produced in the righted electrical and magnetic.		as in electrical current in mass spectroscopy. Jons in solution can be transported, and deposited if desired, under the influence of various applied potentials for consonant for electrical-conductivity measurements. Inherent and induced magnetic properties give most of specialized techniques, such massed to specialized techniques, such massed to specialized techniques, such as acygen analysis based on its paramagnetic properties and nuexeccingly precise and selective for the determination of the compounds of many elements.
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negligible mass and a unit negative charge. The neutrons and protons make up the atomic nucleus. Ordinarily each nucleus is provided with enough electrons to balance the positive charge on it.

In an excited atom the electrons are in an orbital arrangement about the nucleus occupying discrete energy levels, the lowest level being designated 1S and proceeding upward according to the quantum rules. By providing energy in an appropriate form, an electron can be pushed to a higher level. An excited electron exhibits a strong tendency to return to its normal level, and is so doing emits a quantum of electromagnetic radiation.

The number of protons in the nucleus, which fixes the number of electrons determines the chemical and physical properties (except mass which is determined by the number of protons and neutrons) of the resulting atom.

Chemical combinations of atoms into molecules usually involves the loss, gain, or sharing of electrons among the atoms. Every configuration of atoms in a molecule, crystal, solid, liquid, or gas can be represented by a system of electron energy states. Figure 4-4 is an energy level diagram of a typical organic molecule. In an unexcited condition, the molecule can be described as being in a ground state, which is designated S_0 . Two series of electronically excited states can exist in some molecules, the singlet series, designated S_1 , S_2 , ..., S_n , and the triplet series, designated T_1 , T_2 , ..., T_n . A triplet level generally

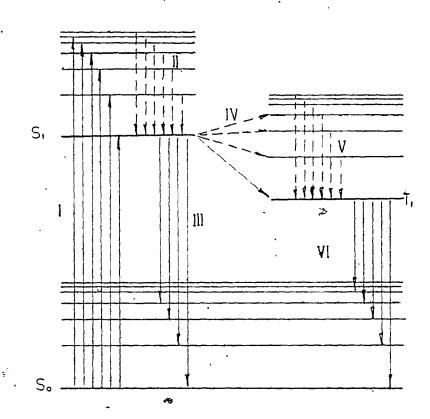


fig. 4-4. Energy-level diagram of a typical organic molecule including only ground singlet, first excited singlet, and its corresponding triplet state. Solid lines indicate radiational transitions, and dashed times indicate nonradiational transitions.

-rocess 1: absorption

11: Vibrational deactivation

111: fluorescence.

1V: intersystem crossing to triplet state

V: vibrational deactivation in the triplet system

V1: phosphorèscence.

has less energy than the corresponding singlet. These two series refer to a difference in the net electronic spin of atoms in the various levels. Because it is difficult to change electron spins, energy transitions as a result of the absorption of radition, raise a molecule only from one singlet state to a higher singlet level or sublevel. The triplet state is reached when it is possible for a non-radiative energy transition from an excited singlet state to the corresponding triplet state to occur within a molecule. The triplet state is metastable while there is a strong tendency for a molecule in a singlet state of excitation to revert back to the ground level.

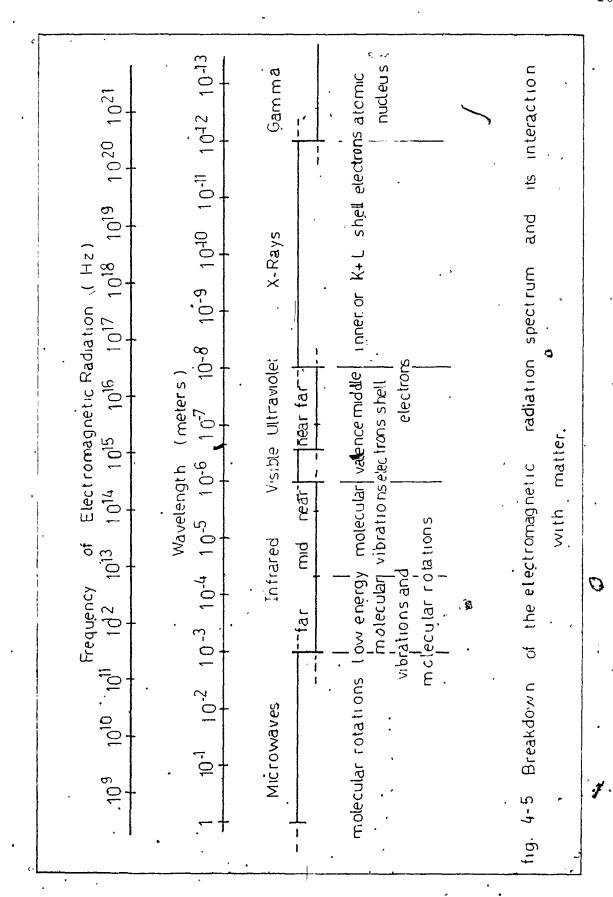
Each electronic energy level, both singlet and triplet has associated with it a series of vibrational sublevels, which correspond to the energy required to excite the various modes of vibration within the molecule. Each vibrational sublevel also has a series of rotational sublevels which correspond to the energy of rotation of various atoms or groups of atoms that make up the molecule.

The four different types of energy and the manner in which they interact with matter are briefly considered below.

1) Electromagnetic radiation is emitted or absorbed by a material whenever there is a change in the energy levels occupied by the electrons associated with the atoms, a change in the mode of molecular vibration, or a change in the rotation of atoms or groups of atoms within the molecule. The electromagnetic spectrum is

broken down into different regions as shown in figure 4-5. The frequency boundaries of these regions are determined by the appropriate practical methods of producing and detecting the radiation. Radiation from the different regions of the spectrum interacts with different "components" of matter, which is also indicated on figure 4-5. X-rays for example which consist of photons of relatively high energy, penetrate deeply into the electron orbits of an atomand possess sufficient energy to push inner electrons to higher energy levels. When such a transition occurs a photon of electromagnetic radiation is absorbed. ever because of their high energy, X-rays, are not adaptable to the causation of lower energy transitions corresponding to pushing a valence electron to a higher energy level or inducing vibration or rotation of intra molecular bonds. Transitions of this nature occur when a material absorbs electromagnetic radiation in the visible:, ultraviolet, infrared, and microwave frequency ranges respectively. The transitions which occur in a material are unique and thus can be used to identify the atoms or molecules present in the sample on a qualitative and a quantitative basis.

Transitions involving atomic or molecular energy levels and electromagnetic radiation can be observed by measuring the selective absorption of radiation passing through them. The frequency at which absorbtion occurs is indicative of which element or molecule is present and the amount of radiation absorbed is indicative of the amound of the element or molecule present in the sample. The energy gained by a material which absorbs



radiation does not remain in that material, but is lost or degraded by several mechanisms. The material loses the absorbed energy by the emission of radiation. Transitions can also be observed by measuring radiation being emitted from a material. It may be emitted as radiation of the same wavelength as that This mechanism is known as resonant fluòrescence. absorbed. Another emission phenomenon, known simply as fluorescence, occur when part of the absorbed energy is degraded to heat, lowering the net energy of the molecule to the lowest vibrational and rotational level within the same electronic singlet level. remainder of the energy is then radiated to return the molecule to the ground state. In the case of fluorescence the persistance of emitted radiation is not measureable. However, in some materials a non-radiative transition from a singlet state to the corresponding metastable triplet state occurs. Because the probability of a transition from a triplet to the ground state is low, radiation may be emitted from the material for some time. This phenomenon is called phosphorescence. The Raman effect is somewhat similar to fluorescence in that the radiation is emitted from the sample with a change in wavelength. The shift in wavelength is caused by extraction of energy from the quanta of incident radiation. The emergent quarta thus can be thought of as the same ones which entered, but with less energy.

2) Chemical Affinity or Reactivity. Chemical reactions contain an inherent ability for the recognition of certain substances.

By mixing, the two parties to the reaction can be brought into close proximity so that the potential driving force toward reaction can come into play. Three methods, falling into this category by which qualitative and quantitative composition determinations can be made are the measurement of the amount of a reactant consumed until an end point is reached, the amount of reaction products that are generated, and the amount of thermal energy liberated during a chemical reaction which is initiated when an appropriate chemical is introduced to the sample.

A method that is currently used to measure the moisture content of green sand in several foundries including General Motors of Canada is done by mixing a sample of sand with an excess amount of calcium carbide powder and measuring the volume of acetylene gas which is generated. The volume is directly is proportional to the moisture content. This technique is typical of the methods in this category in that they are primarily suited for the analysis of batch rather than continuous samples.

methods for the determination of chemical composition can be used when it is possible to rely upon some inherent or conferred electrical or magnetic distinction between the soughtafter components. In order for the distinguishing features to be sorted out and measured the sample must be incorporated in a suitable electric or magnetic circuit. Some of the techniques within this class of methods are mentioned below.

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Mass spectroscopy is capable of sorting out the constituent ions in a gas or vapor sample according to their mass and conferred electric charge. Techniques for the analysis of solids and liquids have also been developed.

There are a number of electrochemical measurement techniques based on the ability of materials to dissociate into ions in solution or in a molten bath and migrate toward electrodes when under the influence of an electric field. It is possible to make both qualitative and quantitative composition measurements using these methods.

There are several methods that are based on the inherent electrical properties of materials. Electrical conductivity is used for the determination of the purity of water. Measurement of the dielectric constant of a material is used for determining small amounts of water in some materials. As mentioned previously the American Foundryman's Society (5) and other have proposed that both of these properties be used to measure the moisture content of green sand.

The magnetic properties of materials are also utilized of for composition measurements. Oxygen and nitrous oxides exhibit paramagnetism which enables them to displace other gases from strong magnetic fields, permitting their determination. Nuclear magnetic resonance which is the name given to a phenomenon involving the interaction of elements possessing nucleii with odd atomic numbers, with a strong homogeneous magnetic field acting in combination with a weaker oscillating field offers a lot of useful

information, including structural determinations and qualitative and quantitative measurements of the composition of materials.

4)., Thermal or Mechanical Energy. The interactions between matter and energy falling into this category, afford information of a less specific nature when compared to many of the methods in the first three categories. For example, some gas molecules possess the distinguishing ability to conduct larger amounts of heat away from a heating element than others. The overall cooling effect can be used to measure the concentration of a particular gaseous element in a mixture of gases. conductivity analyzers designed to operate based on this principle are limited in their specificity or ability to recognize a particular gas. However, according to Considine (15), when such an instrument is to be used in an application where it is necessary to measure a gas with high thermal conductivity such as hydrogen in a gas with low.thermal conductivity such as nitrogen it is possible to obtain accurate results. Another example of a measurement technique of limited specificity in this category is the measurement of viscosity to determine concentration, degree of polymerization, composition, etc. under controlled circumstances along with advance knowledge of all materials present in the sample is it possible to obtain specific results.

Any attempt to use an instrumental technique to measure

a green sand constituent must recognize that there are a number of practical considerations involved which limit their utility.

These considerations are described below.

1) The sample must be representative. According to Willard,
Merritt, and Dean (34) sampling may involve up to ninety percent
of the problem of automatic control.

The requirement that the sample be representative involves the size of the sample and the manner in which it is obtained.

According to Yearley (4) the additives are not uniformly distributed throughout the sand even when mulled as long as 20 minutes. Yearley came to this conclusion as a result of tests using a laboratory type sand muller. In the foundry industry the mulling time of green sand is less than one minute in some instances and frequently between one and two minutes. The moisture, clay, and seacoal constituents of green sand are added and blended with the sand during the mulling operation. The fact that the additives were found not to be uniformly distributed throughout the sand when mulled at least ten times as long as in actual practice indicates that in order to be representative the sample must be of substantial size.

The American Foundryman's Society (38) has done some work on sampling procedures for green sand. They have recommended that the samples be collected in one quart, air tight containers to prevent loss of moisture, exercising caution not to pack the sand. They also recomment that the sample be riddled through a 1/4-inch mesh screen before testing and that actual manual

tests for composition be conducted on 50 gram samples. To give the reader an idea of the size, a 50 gram sample of loose green sand fills a two inch diameter tube to a depth of one inch. actual practice some foundries have departed from the recommendation by the AFS and have devised their own standard for sample General Motors of Canada in St. Catharines conducts moisture tests on three gram samples of green sand. A three gram sample of loose green sand fills a 5/8-inch diameter tube to a depth of approximately 5/8 of an inch. The three gram sample size was selected because it suited an apparatus which ',was available for use at that particular time. Moisture, as mentioned previously, is determined by the gas bomb technique. This method was used in parallel with the standard method of oven drying a 50 gram sample until the sand laboratory supervisor was satisfied that the two methods produced similar results. The actual data that enabled him to come to this conclusion has subsequently been lost. At General Motors other constituents are measured from 50 gram samples as recommended by the AFS. The writer is of the opinion that a 50 gram sample is large \cdot enough to ensure proper representation in all cases while the , three gram size could easily produce erroneous results. writer bases his opinion on the findings of Yearley (4), the fact that the mulling times that he used were at least 10 times longer than those encountered in actual practice, and the fact ... that small core butts which are often present in green sand , would have a greater effect on a small sample than a larger one.

For the purposes of automatic control the amount of sand in the measurement chamber, being subjected to energy from an external source should weigh no less than 50 grams.

A comprehensive study is needed to determine the minimum representative sample size not only for the measurement of moisture but for the clay and seacoal constituents as well.

Information of this type is important because some analytical techniques require that the sample be smaller than even the three grams of green sand.

The AFS also has a recommendation concerning the manner in which a sample of sand is obtained, namely that when a sample is taken from a sand pile or storage bin, it be from a depth of not less than six (6) inches below the surface of the sand and when a sample is taken from a moving conveyor that it be immediately down stream of the aerator. This is to ensure that moisture losses due to evaporation be kept to an absolute minimum.

2) A second practical consideration concerns the physical state of the sample. Once the proper method of obtaining the sample and the sample size has been established, the measurement technique selected must enable the external energy source to interact with the entire sample and that the result of the interaction be subject to observation. For many materials this is not possible The majority of analytical methods are applicable to gases and vapours, fewer to liquids, fewer still to solids, and fewest to particulate or granular solids. When compared with a solid, the molecules of a gas are widely spaced and freer to react with

energy from an external source in a characteristic manner. The reference work by Considine (14) includes a rather exhaustive table, describing a total of 104 analytical techniques. Of the 104, only 13 are applicable for the analysis of a particulate solid sample, some of them offering information not in line with the objective of measuring the composition of green sand. Examples of techniques which cannot be used for the analysis of agranular solid, are the methods involving the transmission of infrared, visible, and ultraviolet electromagnetic radiation because green sand is opaque to the radiation. X-radiation, on the other hand, having greater penetrating power can be transmitted through and interact with a solid sample. The fact that green sand is a granular solid limits the number of techniques that can be used for analyzing its composition.

specificity of the method. It would be desireable to have analytical methods which are specific for each element or perhaps each class of compounds. A specific method for an element would permit its identification and measurement in any material. Unfortunately very few methods are completely specific. According to Ewing (27), it is sometimes necessary, during the course of the laboratory analysis of a sample, to isolate the desired constituent in a measureable form or to remove interfering substances from the sample.

An example of an analytical method that, to a certain extent is specific for many metallic elements is atomic absorption.

This method involves dissociation of a compound into its element in a flame. In the flame the atoms are able to absorb radiation from an external source which, as mentioned previously, is then emitted. Both the incident and the emitted radiation are of the same frequency. Unfortunately atomic absorption is best suited for analysis where the sample is a liquid solution so that it can be introduced into the flame in the form of a mist. Thus it is not adaptable for analyzing green sand.

The point is that sometimes a method for the measurement of a certain constituent cannot be used if other constituents, present in the sample, interfere with the measurement or have similar interactions with the energy from the external source. A fourth practical consideration was first mentioned in the third chapter. It is desirable that the analytical method be non-destructive. It is evident, from an examination of table 4-1, that many analytical techniques are of a destructive nature. Included are the following: emission spectroscopy in which the samples are thermally excited to emit radiation, the methods, of the second group in which the sample is mixed with a reactant, mass spectroscopy in which a solid sample can be analyzed only. if it is vaporized or pyrolyzed to give characteristic fragments, and the methods of group four (determination of melting point, boiling point, and vapour pressure) in which the sample is subjected to heat.

Destruction of the sample creates a problem regarding disposal of the sand residue. If the residue was discharged back

into the sand, a build-up would result because the sand is recycled. Depending on what the residue consisted of this could cause a problem with the sand. If the method selected involves the addition of a reactant to the sample, this also introduces the problem of handling this material.

5) A final practical consideration concerns an operating characteristic of the analytical technique. Instruments for measuring process variables may be described as periodic or continuous. Periodic instruments are generally automated versions of conventional laboratory equipment. Instruments for the determination of moisture using oven drying, chemical titrations, and distillation methods are available in automated form, with a measurement typically requiring two minutes or longer. According to Considine (15) the periodic type of instrument is quite impractical for automatic systems.

*The practical considerations involved in the selection of an analytical technique to measure the composition of foundry green sand, limit the number of methods that the author can suggest for further work.

X-ray flourescence and gamma ray spectroscopy from group I on table 4-1 appeared to be useful for this application. Considine (15)' describes the use of X-ray flourescence to measure aluminum. Further work is required to determine if this method could be used to measure clay content which is the only constituent in green sand which contains the element aluminum. Gamma ray spectroscopy, which is also known as Mossbauer spectroscopy, is

a study of the phenomenon of resonance fluorescence of gamma radiation involving intra nuclear rather than electronic energy levels. Most of the research in this field has been with the isotopes ⁵⁷Fe and ¹¹⁹Sn. However, according to Cohen (41), it is unlikely that lighter elements, such as those in the make up of green sand, will ever be analyzed by the Mossbauer method. The energy of gamma radiation required to excite isotopes lighter than ⁵⁷Fe is low enough for the Compton effect to occur. The Compton effect takes precedence over the Mossbauer effect at these frequencies with the result that most of the radiation will be absorbed by the electronic interaction. The Mossbauer effect is therefore not observable for the light elements which make up green sand.

The methods of group II on table 4-1 are intended for use on batch samples rather than continuous samples. The samples are also destroyed or altered in some way. The author does not recommend the use of these methods for the continuous measurement of green sand composition.

The methods of group III on table 4-1 that appear to have potential for this application have already been tried by others. These methods cannot be used either because the repeatability does not lend itself to this application or because of the presence of other constituents that interfere with the measurement method.

The methods in the fourth grouping on table 4-1 are not applicable for the measurement of the composition of green sand.

These methods are best suited for the analysis of materials in

the gaseous or liquid state.

C) Results of Literature Survey.

The analytical methods for the measurement of the composition and properties of green sand discussed in this chapter are described on table 4-2. Most of the methods for composition measurement are not suitable for this application for at least one of the following reasons.

- 1. The technique is periodic in nature and as such is not compatible with automatic control systems.
- 2. The repeatability of the technique is not adequate for the application.
- Other constituents that interfere with the measurement method are present.

There are several techniques that require further evaluation.

These are listed below.

- 1. The infrared moisture measurement method has been subjected to a foundry trial on two occasions. On both occasions the instrument did not respond to changes in the sand moisture content. Further work is required to identify the exact reason for the response failure. The instrument which makes the measurement is compatible with automatic control systems.
- 2. X-Ray fluorescence, which can be used to measure the amount of aluminum in a material, is suggested as a method to measure clay content, which is the only constituent in green sand that contains aluminum. Further work should be to determine if the

method is adaptable for continuous measurement and to determine if the repeatability is adequate for this particular application.

- 3. Neutron **Scattering** which measures the amount of hydrogen in a material, could be used to measure the seacoal content of green sand, provided that a method to measure moisture content is found.
- 4. According to Vingas (42) the active clay content of foundry green sand can be determined with the use of the compactability test provided that the sand moisture content is measured by some other method. The compactability test is currently a manual method. The author is of the opinion that it could be developed into a continuous device. A sketch of a continuous compactability measuring device is shown in fig. 4-6. Sand from a muller is riddled through a screen onto a small conveyor. The loose sand is compacted between the lower conveyor and an upper conveyor which is squeezed against the lower conveyor by an air cylinder. The compactability is measured with a displacement transducer.

It is apparent that, when a composition measuring system is eventually developed it will be necessary to measure the density and possibly the temperature of green sand. These two variables can be measured with a high degree of precision by the use of the gamma ray absorption method and by the use of resistance temperature devices.

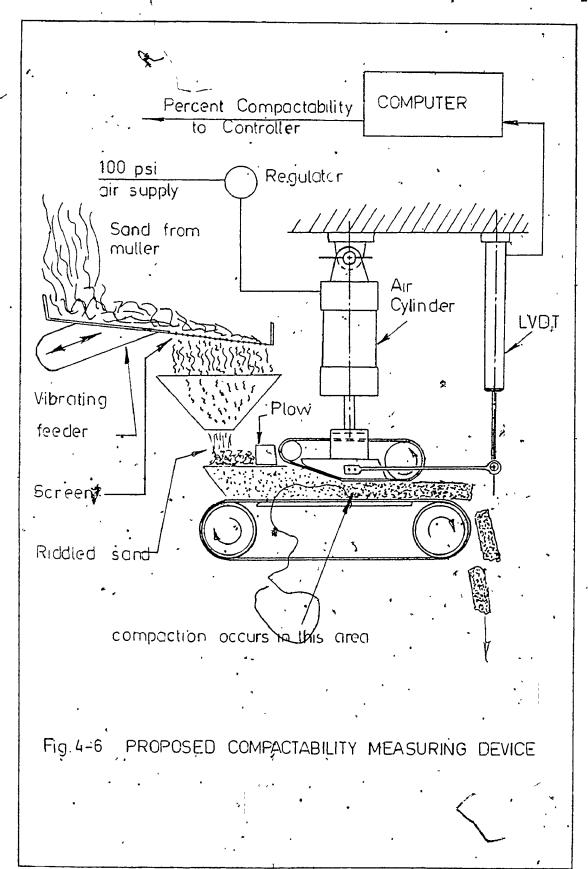


TABLE 4-2. Methods to Ascertain the Composition and Properties of Green Molding Sand

	4			1	
Constituent or property to be meas-	Name of Analytical Technique	Nature of Technique	Åocuraey	Repeat- ability	Remarks
I Moisture	Drying &	Periodic	data not available	data not available	This method is used as a standard to calibrate other instrument techniques. Impractical for use on automatic control systems.
. 2	Ġas Bomhb	Periodic Sample is destroyed	data not available	data not. available	Impractical for use in automatic control systems.
e	Electri- cal cond- uctivity	Periodic or conti-	+0.25% water under controlled conditions	affected by clay, seacoal & coke content temperature & density	Repeatability is inadequate for this application
4	Humidity Sensing	Periodic . or conti- nuous	+2.0% water	data not available	Not suitable for this application. Equilibrium between relative humidity of air and moisture content of green sand does not exist. The method has a slow response to changes in moisture.
·v	. Capacit- ance	Periodic or conti- nuous	+0.5% .water	data not available	Repeatability is inadequate for this application.
	1				

		_			
9	Multiparameter method	Periodic or Continuous	between +0.50 & +0.80 % water under controlled conditions	affected by variables similar to electrical conductivity & capacitance methods	Repeatability is not expected to exceed that obtainable by electrical Conductivity or Capacitance methods. Therefore it is inadequate for this application.
	Neutron Absorp- tion	Perjodic or Continuous	+0.25% water	repeatability affected by the sea-coal content	Cannot be used unless a good method of measuring the seacoal content is available (See item 111-2).
ω	ጀ አ	Periodic. Possibly could be used as a continuous device	+0.03% water	data not avail- able	Presence of iron in the sample interferes with the measurement of moisture. The technique can therefore not be used.
6	Microwave absorption	Periodic'or continuous	+0.30% water	+0.30% water	The repeatability is not good enough for this application.
0.	Infrared Absorption	Periodic or. Continuous	+0.1% in some applications	+0.1.% in some applications	Method needs further evaluation. Has been reported not to work. Reasons for failure of method are not known. Compatible.with automatic control systems.

Table 4-2 (contd.)

	This method has been adopted as. AFS standard. It measures all fine material which introduces error. It is impractical for use on automatic control systems.	Impractical for an automatic control systems.	Impractical for automatic control systems.	A suggestion for further work. Reference (15) indicates that this method can be used to meagure Al which is an elemental constituent of clay.	The method is impractical for use in automatic control systems. The method is an AFS standard for measurement of seacoal content.	This method does not measure composition. This method augments existing manual control procedures Does not reduce manpower requirement. Incorporation of the method has potential for improved product quality.
•	not available	not available	not available	data not available	data not available	data not available
	not available	not available	not available	data not available	data not avail- able	data.not avail- able
	Periodic Sample des- troyed	Periodic Sample des- troyed	Periodic Samplé des- troyed	Periodic with potential for development into a continuous device	Periodic. Sample is destroyed	Continuous
	Elutriation .	Methylene Blue	Nickel Sulphate	X-Ray Fluores cence	1 Combust- ion	Moldability Controller
	II Clay		m	4	III Sea- coal	IV Mold- ability

Table 4-2 (contd.)

data not Further work is required to develop available a continuous compactability transducer. Along with a continuous moisture measurement method it could be used to control clay content (see ref. 42).	better than +1.0% Compatible with automatic control systems. Repeatability is more than adequate.	+0-1°F Compatible with automatic control systems. Repeatability is more than adequate.
data not available	Between +1.0% better & +0.03% . than +	. 1.00F
Periodic but has potential for development into a contin- uous type device	Continuous	Continuous
V Compact- Compactability ability test	Gamma Ray Absorption	Resistance Temperature Device
V Compact- ability	. VI Density	VII Temper- , ature

CHAPTËR 5

MULTIPARAMETER DETECTION OF MOLDING SAND ADDITIVES

A. Introduction

The multiparameter method of measuring the content of foundry molding sand additives was introduced in Chapter four of this report. The method is described in a paper by Pustynnikov and Protod'yakonov (40). It differs from the other electrical methods of monitoring the composition dn; that the additive contents are not determined from a single criteria such as conductivity, dielectric permeability, or dielectric losses but from a combination of these. investigators described the method as having the capability of monitoring the clay, water, and density simultaneously. The operation depends upon the fact that each variable (water, clay, and packing density) has a certain characteristic frequency at which the current response of the detector is more dependent on it than on any other variable. The actual measuring device consisted of a pair of parallel copper plates arranged in a manner such that the sand could be fed between Three alternating current signals, each one corresponding to the characteristic frequency of one of the variables, were applied to the measuring device. The current

response due to each signal was displayed on a meter graduated in "weight percent water" or "weight percent clay" after the effects of fluctuation in the other variables were balanced against it. An instrument, based on this principle of operation, is reported to have been built and tested in a foundry in the Soviet Union. According to the literature it produced satisfactory results. This method had to be evaluated experimentally because the results from actual foundry proving trials to support the contention that the method actually worked satisfactorily were not included in the paper. This evaluation is described in the next section of this chapter.

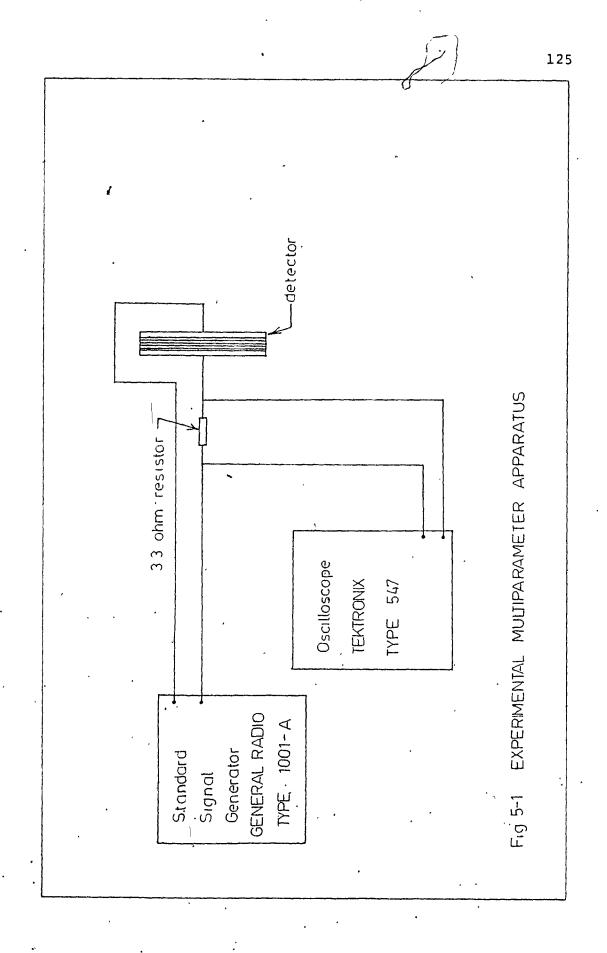
- B. Experimental Objectives, Procedure, and Apparatus

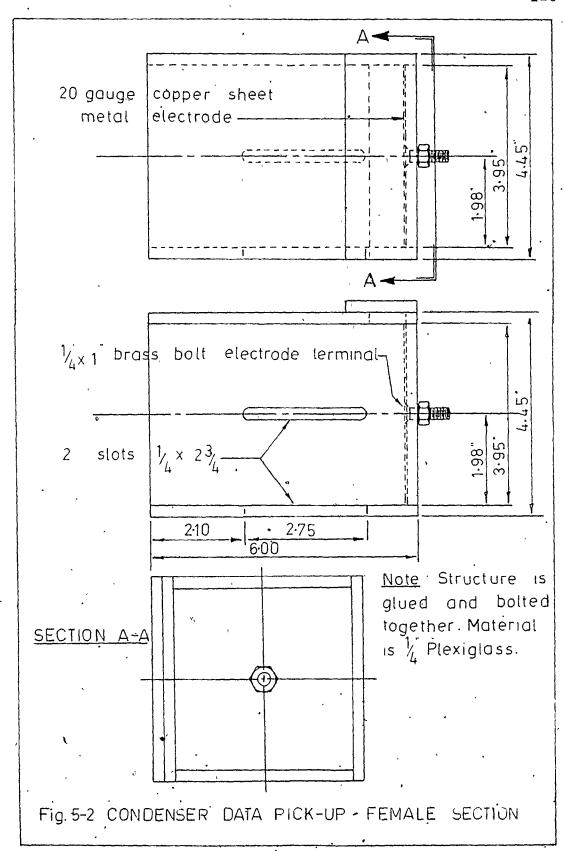
 The experimental objectives were three-fold.
- 1) The first objective was to find the characteristic frequency of each variable.
- 2) A second objective was to determine the effect of adjusting the level of each variable on the current response at its characteristic frequency and at the characteristic frequencies of the other variables.
- 3) It was reported that additional adjustments to the current response of each measured variable at its characteristic frequency would be necessary if other components such as seacoal were present in the sand. Hence the third objective was to find a characteristic frequency for seacoal and to

find the effect of changing the seacoal content on the current response at the characteristic frequency of each other variable.

In order to meet these objectives, molding sand samples, in which each additive was varied independently, were tested in a capacitor-type detector which consisted of two square copper plates held parallel to each other in a plexiglass frame which is shown in figure 5-2, 5-3, The frame was constructed in such a manner that the spacing between the copper plates could be adjusted. The frame had an opening in the side with a removable lid to allow easy loading of molding sand into the container and to prevent moisture from escaping during the test. Due care was exercised during the loading so that each sample of sand tested was of the same weight. Equipment used along with the detector was a Tektronix Type 547 Oscilloscope and a General Radio Type 1001-A Standard Signal Generator. This equipment was connected utilizing shielded cable to reduce noise pick-up as shown on figure 5-1. A photograph of the actual experimental set-up is shown in ·figure 5-5.

In order to make the mixture uniform it was necessary to mix the molding sand ingredients into the molding sand in a certain order. The uniformity of the mixture was acceptable if the molding sand was mixed in the order given below.





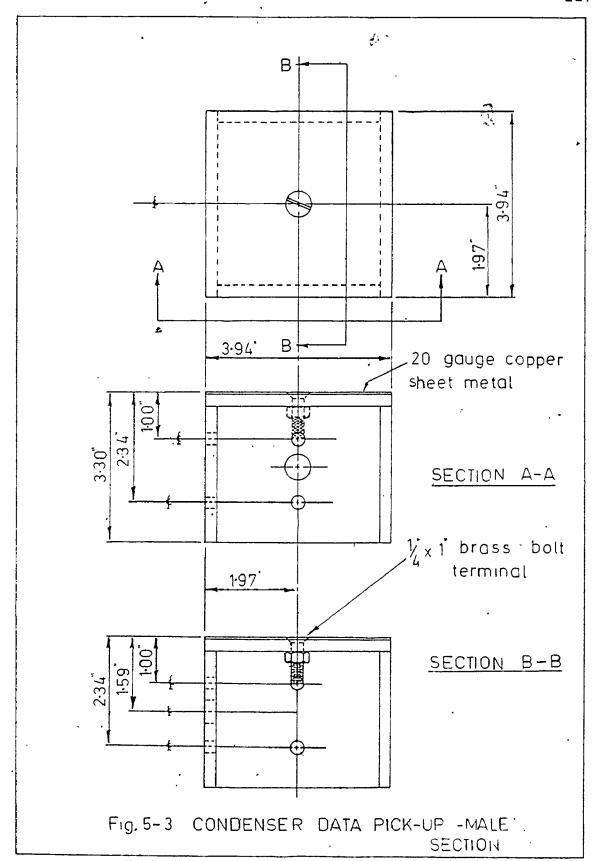


figure 5-4 Condensor: Data Pickup

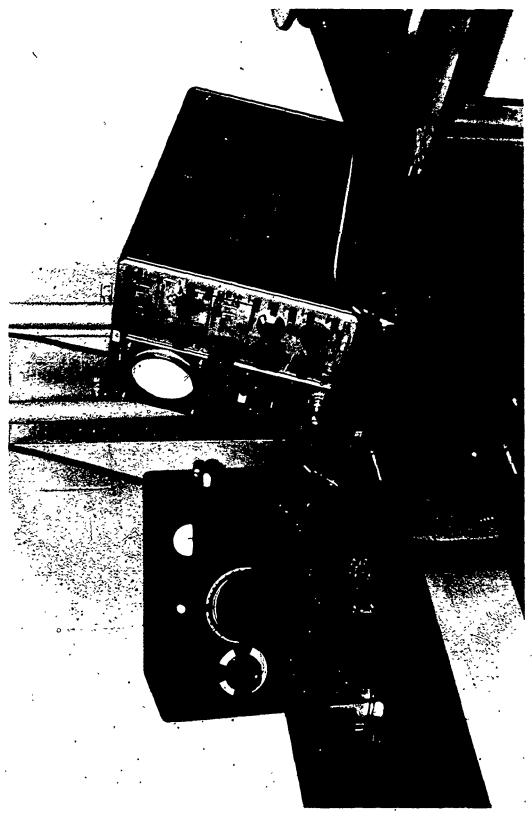


figure 5-5 Multiparameter Monitoring Experimental Set-up

- i) Table 5-1 listing the composition of each sample was consulted and the appropriate amount of silica sand was placed in a container.
- ii) The water was stirred in with the sand grains until they were wetted thoroughly.
 - iii) The seacoal was added and mixed in.
- iv) The clay constituent which was thirty percent western bentonite and seventy percent southern bentonite was added last.

When a sample was mixed in some other sequence the clay had a tendency to ball up making it impossible to coat the sand grains evenly.

At a detector plate spacing of one centimeter, a one hundred and twenty gram loosely packed sample could be placed into the opening between the plates.

A two hundred gram batch of each sample described on table 5-1 was prepared and tested. The packing density was varied by forcing more sand into the opening. The frequency of the applied voltage and the corresponding current through the detector were recorded in tabular form at predetermined frequencies over the range of 0 to 50 Mhz. The level of the applied signal was 0.2 volts peak to peak (this was a machine limitation). The voltage drop across the measuring resistor, rather than the actual current through the detector was recorded because the voltage drop is related to the

Table 5-1 Composition of Molding Sand Samples Tested by Multiparameter Technique

				 	
Sample Number	Water Content	Seacoal Content	Clay Content	Silica Sand Grains	Packing Density
1 2 3 4 5 6 7 8	2.00 2.60 2.70 2.80 2.90 3.00 4.00 5.00	5.00 5.00 5.00 5.00 5.00 5.00 5.00	9.50 9.50 9.50 9.50 9.50 9.50 9.50	83.50 82.90 82.80 82.70 82.60 82.50 81.50 80.50	1.20 1.20 1.20 1.20 1.20 1.20 1.20
9 10- 11 12 13 14 15	2.80 2.80 2.80 2.80 2.80 2.80 2.80 2.80	4.00 4.70 4.85 5.00 5.15 5.30 6.00 7.00	9.50 9.50 9.50 9.50 9.50 9.50 9.50	83.70 83.00 82.85 . 82.70 82.55 82.40 81.70 80.70	1.20 1.20 1.20 1.20 1.20 1.20 1.20
17 18 19 20 21 22 23 24	2.80 2.80 2.80 2.80 2.80 2.80 2.80 2.80	5.00 5.00 5.00 5.00 5.00 5.00 5.00	8.00 9.00 9.25 9.50 9.75 10.00 11.00	84.20 83.20 82.95 82.70 82.45 82.20 81.20 80.20	1.20 1.20 1.20 1.20 1.20 1.20 1.20
25 26 27 28 29 30 31 31	2.80 2.80 2.80 2.80 2.80 2.80 2.80 2.80	5.00 5.00 5.00 5.00 5.00 5.00 5.00	9.50 9.50 9.50 9.50 9.50 9.50 9.50	82.70 82.70 82.70 82.70 82.70 82.70 82.70 82.70	1.00 1.10 1.15 1.20 1.25 1.30 1.40

S.

current by a constant and the voltage drop could be read directly from the oscilloscope. The voltage drop across the measuring resistor is herein referred to as the current response of the detctor or more simply as the current response.

Several measures were taken to reduce the scatter in the data. These are listed below.

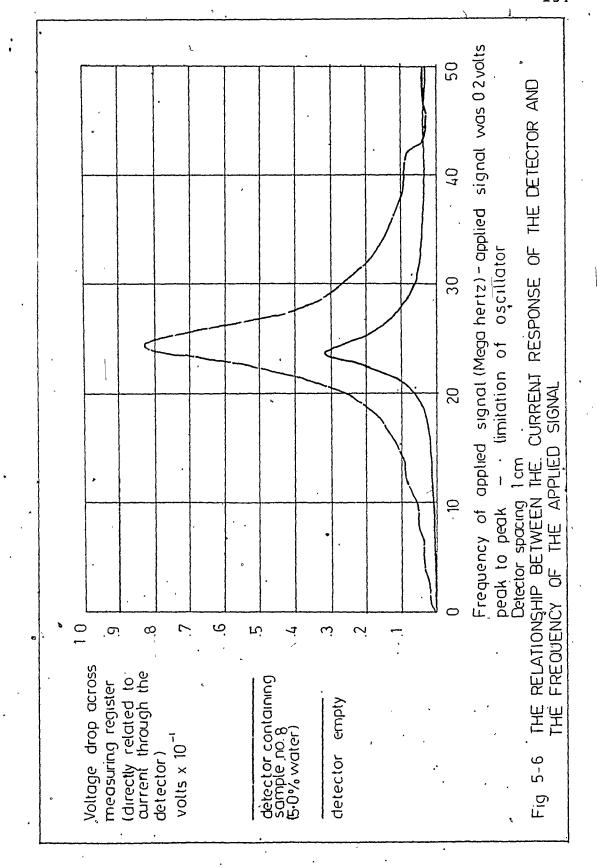
- a) The constituents of each batch of sand were individually weighed prior to mixing, accurate to 0.05 grams.
- b) The use of a constant temperature and humidity room and the adoption of a standard time for mixing and loading the sample into the detector were to equalize evaporation losses of water, the only volatile component in the sand mixture, for each sample.
- c) Enough rigidity was incorporated into the design of the detector to prevent the copper plates from being forced apart when samples of high packing density were tested.
- d) The detector plates were cleaned after each sample was tested.

C. Experimental Results

The current response, at a number of different frequencies, over the range of 0 to 50 Mhz, was recorded in tabular form, for each of the samples described in table 5-1. The complete experimental results are included in the appendix of this report. The data from each sample when plotted on

graph paper and joined with a smooth line as in figure 5-6, forms a bell shaped curve. This is to be expected because the circuit (see figure 5-1) is a resistance, a capacitance (detector), and an inductance (any circuit has some inductance) in series. According to Skilling (43) this type of electrical circuit has a relatively low impedance to current, over a certain frequency range because the inductive and capacitive effects cancel each other, making the circuit look as if it were merely resistive. This phenomenon, known as resonance, gives rise to the bell shaped curve.

In the case of the samples in which seacoal and clay were varied, the current response was approximately the same over the entire frequency range, even when the clay and seacoal contents were adjusted substantially beyond the normal operating ranges in molding sand. Table 5-3 shows the seacoal content of samples 9 through 16 (Table 5-1) and the corresponding current responses at four different frequencies. This information is displayed graphically on figure 5-8. The least squares straight line was drawn through the data corresponding to each frequency. The choice of the four frequencies was to have been based on the proposition that each variable has a characteristic frequency at which the current response is more dependent on its presence than on any other variable. However, examination



of the data showed that the seacoal content did not affect the current response at any frequency between 0 and 50 Mhz.

Table 5-4 and figure 5-9 gives similar information for the clay constituent. Again a characteristic frequency was not evident from the data.

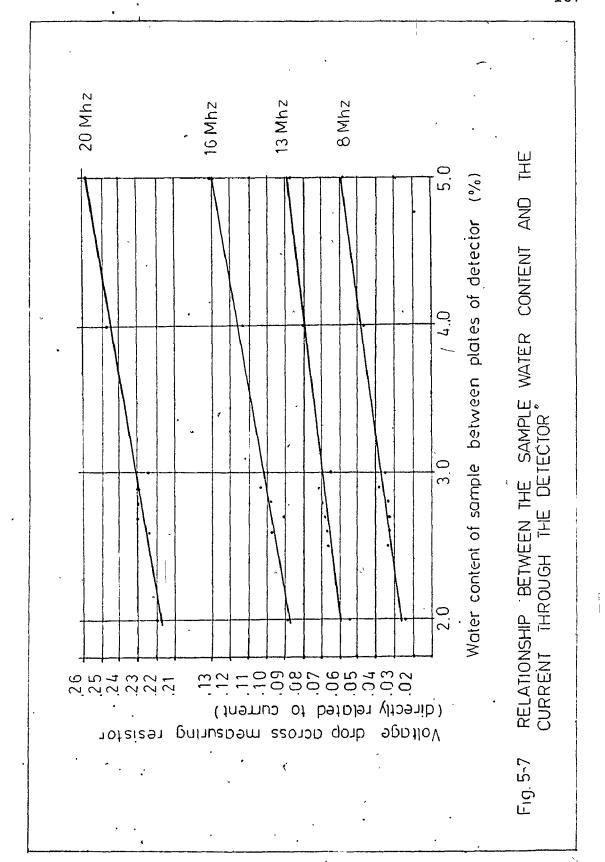
In the case of water content and packing density, it was found that the current response increased as these variables increased. Table 5-2 and figure 5-7 show the effect that the water content has on the current response at the same four frequencies used previously. straight line due to the method of least squares was drawn through each set of data points. Table 5-5 and figure 5-10 show similar information for the packing density. While the current response increased as the water content and packing density increased, the characteristic frequencies, described by Pustynnikov and Protodyakonov, at which the effect of water content and packing density upon the response are particularly pronounced, were not evident from the data. The water content and packing density are related to each other as well as to the current response. A fluctuation in each variable results in a change in the total amount of water inside the detector. With reference to table 5-1, the samples numbered 1 through 8 and 25 through 32, which pertain to the effect of water content and packing density upon current response, were arranged in table 5-6 so that the total water

Table 5-2 The relationship between the voltage drop across the measuring resistor and the sample water content at four different frequencies:

Sample	Water	Voltage	drop acros	ss measurin	ng resistor
Number	Content	8 Mhz	13 Mhz	16 Mhz	20 Mhz
1 2 3 4 5 6 7 8	2.00 2.60 2.70 2.80 2.90 3.00 4.00 5.00	0255 .0335 .0335 .035 .0385 .036 .048	.056 .068 .069 .072 .063 .079	.088 .097 .090 .098 .104 .100	.220 .225 .230 .230 .230 .225 .248

Table 5-3 The Relationship between the voltage drop across the measuring resistor and the seacoal content at four different frequencies

Sample	Seacoal	Voltage	Voltage drop across measuring resistor				
Number	Content	. 8 Mhz	13 Mhz	16 Mhz	20 Mhz		
9 10 11 12 13 14 15	4.00 4.70 4.85 5.00 5.15 5.30 6.00 7.00	.034 .037 .039 .0350 .0335 .038 .036	.065 .068 .071 .069 .065 .070	.096 .100 .103 .098 .098 .102 .099	.210 .232 .235 .230 .230 .230 .228		



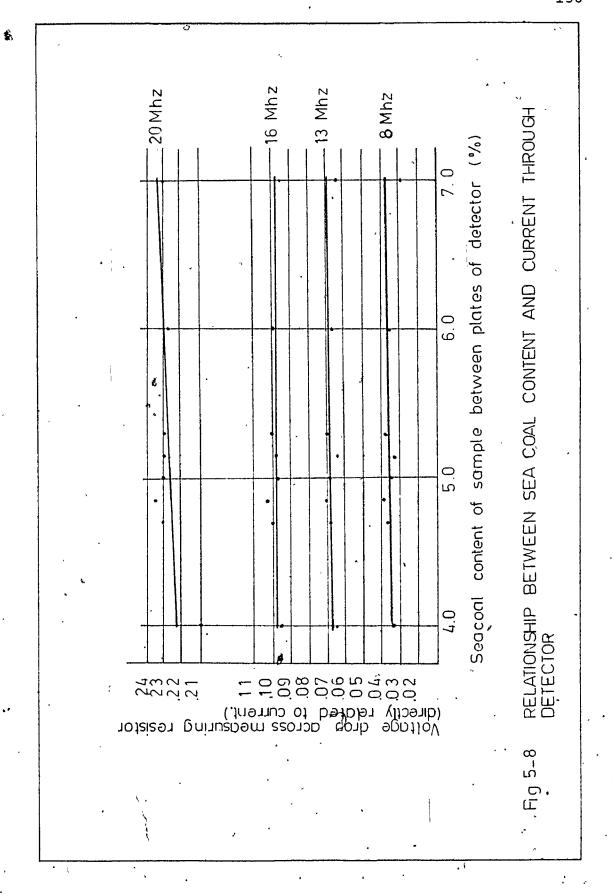
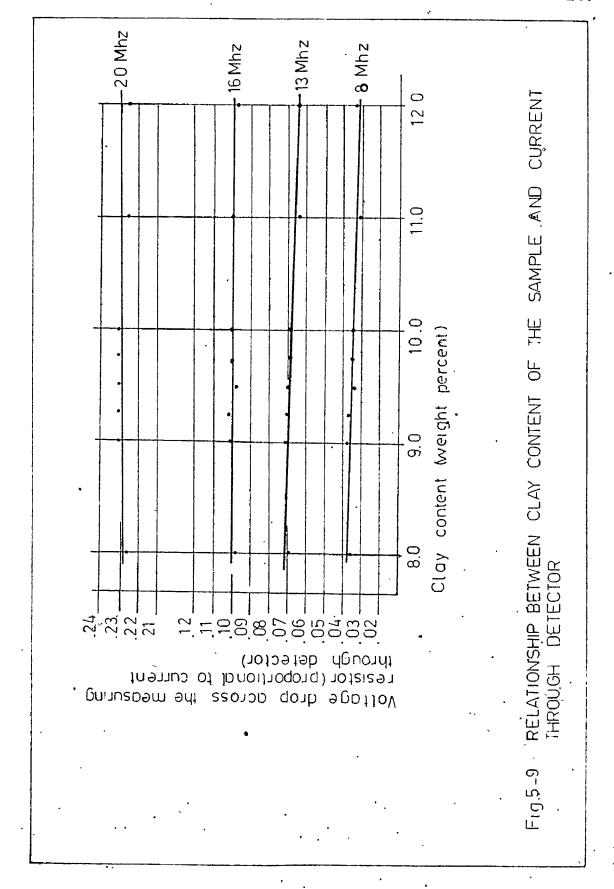


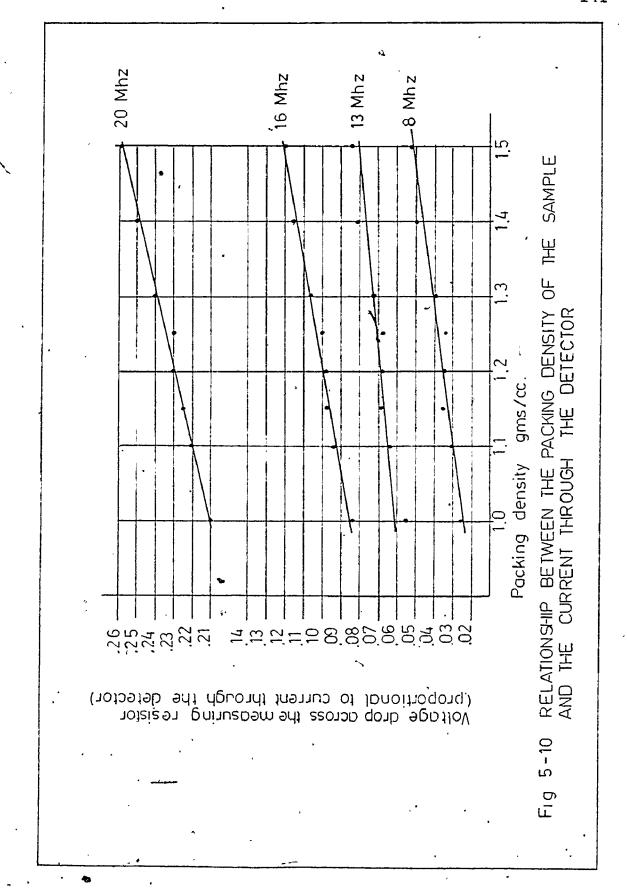
Table 5-4 The Relationship between the voltage drop across the measuring resistor and the clay content at four different frequencies.

Sample Number	Clay Content	Voltage drop across measuring resistor			
Number	concent	8 Mhz	13 Mhz	16 Mhz	20 Mhz
17 18 19 20 21 22 23 24	8.00 9.00 9.25 9.50 9.75 10.00 11.00	.036 .0375 .037 .035 .035 .035	.068 .070 .070 .069 .068 .068	.097 .100 .101 .098 .100 .100	.226 .230 .230 .230 .230 .230 .226

Table 5-5 The Relationship between the voltage drop across the measuring resistor and the packing density at four different frequencies.

Sample Number	Packing Density	Voltage drop across measuring resistor			
Number	voliber Density		13 Mhz	16 Mhz	20 Mhz
25	1.00	.0255	.0555	.084	.210
26	1.10	.0310	.0640	.094	.220
27	1.15	.035	.068	.098	.225
28	1.20	.035	.069	.098	.230
29	1.25	.034	.068	.100	.230
30	1.30	.038	.073	.106	.240
31	1.40	.050	.082	.115	.250
32	1.50	.0525	.084	.120	.255





inside the detector increases. The current response for each of these samples at the four different frequencies is also given. This same information is displayed graphically in figure 5-11. The best least squares straight line is drawn through the data corresponding to each frequency. The vertical distance from the least squares line, to the parallel lines on either side of it, is the standard error of estimate of the current response on the total weight of water inside the detector (see Appendix Page 192). The shaded area contains approximately 68 percent of the sample points.

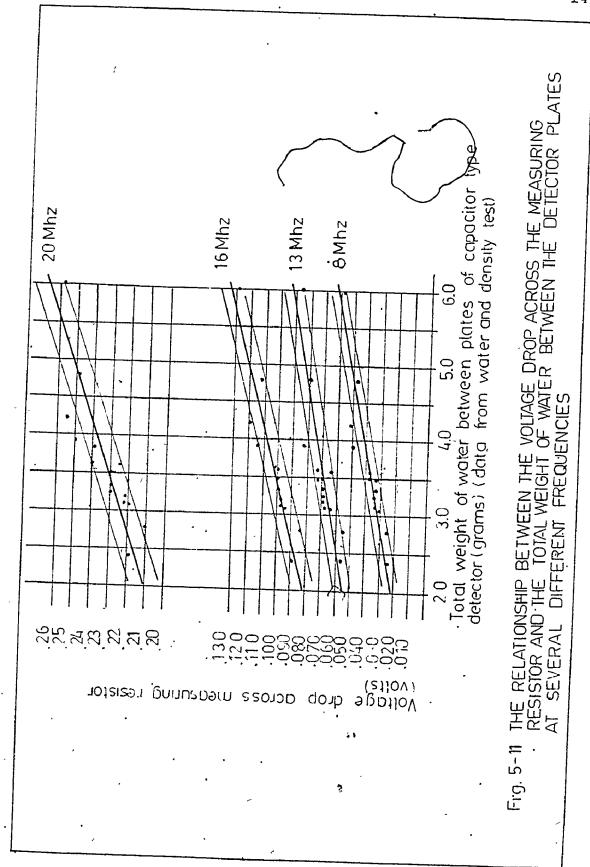
The fact that the data pertaining to water content and packing density can be made to fall along the same line, indicates that the current response is sensitive only to the total weight of water inside the detector. If this method were to be used in an industrial control system, the density would have to be measured by some other means.

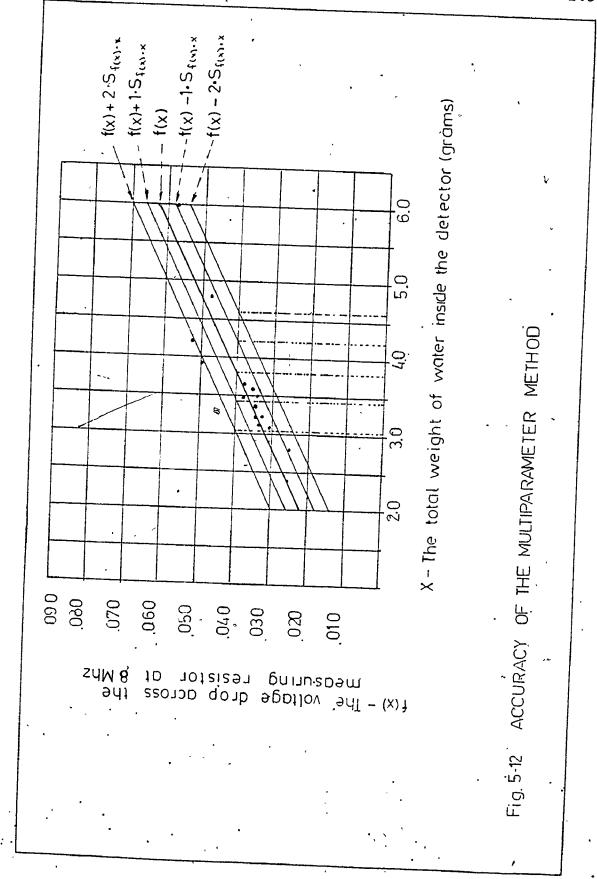
In addition to this limitation, there is also an accuracy problem. Figure 5-12 is an enlarged reproduction of the data corresponding to 8 Mhz shown on figure 5-11. This data set was selected because the ratio of the standard error of estimate to the change in response for a given change in the weight of water inside the detector was lower than for the other three frequencies. The least squares straight line through the data points on figure 5-12 is represented by the equation f(x) = 0.0016 + 0.0101 x. The standard error of estimate of f(x) upon x, F(x).x, was found to be 0.00386.

Table 5-6 The relationship between the voltage drop across the measuring resistor and the total weight of water between the detector plates of four different frequencies.

Sample Number	Water Content	Density	Weight of Water	Volta		across m	easuring
	8	gm/cc	gm	8 Mhz	13 Mhz	16 Mhz	20 Mhz
1 25 26 2 27 3 4 & 28 5 29 6 30 31 32 7	2.00 2.80 2.80 2.60 2.80 2.70 2.80 2.90 2.80 3.00 2.80 2.80 4.00 5.00	1.10 1.20 1.15 1.20 1.20 1.25 1.20 1.30 1.40	2.40 2.80 3.08 3.12 3.22 3.24 3.36 3.48 3.50 3.60 3.64 3.92 4.20 4.80 6.00	.0255 .0255 .0310 .0335 .035 .035 .0385 .034 .036 .038 .050	.056 .0555 .0640 .068 .068 .069 .072 .068 .063 .073 .082 .084 .079	.088 .084 .094 .097 .098 .090 .098 .104 .100 .106 .115 .120	.220 .210 .220 .225 .225 .230 .230 .230 .230 .255 .240 .255 .248 .258

(data from water and density test)
 (sample numbers refer to table 1)





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These calculations are found in the appendix on page The area between $f(x) - 2 f(x) \cdot x$ and $f(x) + 2 f(x) \cdot x$ contains approximately 95% of all the data points. Thus, when a current response of 0.040 is obtained there is a 95% probability the detector is between 3.00 grams and 4.60 grams.

Based on a density of 1.5 gms/cc, which approaches the maximum density to which sand can be rammed, this could be expressed as a 95% probability that the water content is 2.539. ± 0.539. Based on a 100 gram sample, which approaches the minimum density to which molding sand can be riddled, it would be expressed as a 95% probability that the water content is 3.80% ± 0.80%. Even if the density could be measured by some other method with complete accuracy, the best accuracy obtainable would be about ± 0.5% by weight water. This is not accurate enough to control the moisture in molding sand.

D. Summary

The experimental evidence indicates that the multiparameter method could not be used in the manner described by Pustynnikov and Protod'yakonov because:

- 1) none of the variables (clay content, seacoal content, water content, or packing density) exhibited a characteristic frequency.
 - 2) the clay and seacoal contents had, no noticeable

بر effect on the current response.

- total weight of water inside the detector. The relationship between the current response and the total weight of water inside the detector was observed to be linear. Because a fluctuation in either the water content or the packing density affects the total weight of water, another technique would have to be used to measure density if the multiparameter method were to be used to control the water content.
- 4) the accuracy obtained does not meet the requirements for a system to control the moisture content of foundry ing sand.

The multiparameter method is not a suitable measurement method for use in an automatic control system to regulate moisture or any of the other constituents in foundry green molding sand.

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

The foundry green molding sand system is an example of a production process—that does not easily lend itself to automatic composition control. The project was originally taken on because of the potential for reduction in labour cost as delineated in the introduction of this report, and for improvement to product quality. A control system such as envisaged in this report would be applicable only to large continuous sand preparation systems. Existing manual control techniques are more than adequate for small jobbing foundries which normally use batch sand preparation systems. As a result this type of control system would be useful to about 50 foundries in North America. These 50 foundries have approximately 120 continuous sand systems in operation.

Therefore the potential sales for such a system is approximately 120.

The majority of this work involved the selection of suitable transducers to measure the amount of each constituent present in the sample. The following methods which were investigated, appear to have no potential for this particular application. The reasons for arriving at this conclusion have been summarized on table 4-2.

- 1) Moisture Measurement Techniques
 - a) Drying and Weighing
 - b) The Gas Bomb Technique
 - c) Electrical Conductivity
 - d) Humidity Sensing
 - e) Capacitance 1
 - f) Multiparameter Method
 - g) Neutron Scattering
 - h) Nuclear Magnetic Resonance
 - i) Microwave Absorption
- 2) Clay Measurement Techniques
 - a) Elutriation
 - b) Methylene Blue Test
 - c) Nickel Sulphate Test
- 3) Seacoal Measurement
 - a) Combustion Technique

Although it has not been possible to obtain a specific design or solution to this problem, it is felt that the work undertaken has been worthwhile in that the problems involved have been defined in considerable detail, and that this project will provide a good background for future investigators.

In particular the following is recommended for further investigation.

1) The infrared absorption technique has been used to measure the moisture content of other materials. The repeatability

has been reported to be better than plus or minus 0.1 percent water for the range of 0 to 10 percent moisture content. The method has been subjected to a foundry trial on two known occasions. These trials indicated that the method did not work. However, further work is required to discover the reasons for the equipment failure.

- 2) It is recommended that the x-ray fluorescence technique be subjected to a trial to measure the clay content of green sand. Clay is the only constituent in green sand that contains the element aluminum. Aluminum in other materials has been measured with the use of this technique.
- 3) The potential for measuring seacoal content by the use of the neutron absorption method should be investigated. The use of this method would be contingent upon the development of a good method to measure the moisture content.
- 4) The design of a continuous compactability measuring device should be the subject of a future investigation. It appears that this method could be used to measure the clay content of green sand provided that an accurate moisture measurement method were found. It also appears that this method could be used to assign a numerical value to the muller operators hand squeeze technique.
- 5) A comprehensive study is also needed to determine the minimum representative sample size for the measurement of the green sand constituents. This information will be important for the x-ray

fluorescence method because it can be used with very small samples.

APPENDIX I

CALCULATION TO DETERMINE THE EFFECT OF FLUCTUATIONS IN

SLURRY HEAD ON THE MOISTURE CONTENT (CONTROLLED VARIABLE)

A muller processes approximately 600,000 pounds of sand per hour. The average amount of sand inside the muller during operation weighs 28,000 pounds. The muller is loaded continuously at a constant rate, and unloaded on an on-off basis at regular intervals.

The slurry tank, shown in figure 3-5, has a level control which allows a fluctuation in slurry head from 19.5 to 20.0 feet. The flow is regulated by a valve which is governed by the equation $Q = CA\sqrt{2gH}$ where Q is the flow quantity, C is an orifice constant, A is the area of flow, g is the acceleration of gravity, and H is the liquid head in feet. If all process loads are stable and the return , sand has a moisture content of 0.80 percent by weight, then 2.00 percent moisture must be added at the muller. If the required 2.00 percent moisture is being added at a slurry head of 19.75 feet, which is the average head, then one must determine the amount of moisture added at a head of 19.5 feet and at 20.0 feet to calculate the deviation in flow.

Low - At
$$H_1 = 19.5$$
 feet $Q_L = CA\sqrt{2g}$ $\sqrt{19.5}$ cfs
$$= CA\sqrt{2g} \quad (4.415)$$
 Average - At $H_2 = 19.75$ ft. $Q_{AV} = CA\sqrt{2g} \quad (4.440)$ cfs High - At $H_3 = 20.0$ ft. $Q_H = CA\sqrt{2g} \quad (4.470)$ cfs The deviation in flow is $Q_H - Q_L = CA\sqrt{2g} \quad (4.470)$ the deviation in flow is $Q_{AV} = CA\sqrt{2g} \quad (4.440)$ the flow is $Q_{AV} = CA\sqrt{2g} \quad (4.440)$ the deviation in flow is $Q_{AV} = CA\sqrt{2g} \quad (4.440)$ the flow is $Q_$

The 2.00 percent moisture, added at the muller fluctuates by 1.24% because of variations in slurry head. The controlled variable will also vary as a result. The high value of the controlled variable will be

$$0.8 + 2.00 (-1.0062)$$
 = 2.8124%

and the low value of the controlled variable will be

The controlled variable fluctuates between 2.79% and 2.81% moisture.

APPENDIX II

EVALUATION OF THE MULTIPARAMETER METHOD - EXPERIMENTAL DATA

(refer to Table 5-1)

·ı

Sample Composition (percent by weight)

Water - 2.0 Clay - 9.5 Seacoal - 5.0

Density 120 grams/cc

Oscillator Frequency	Instrument Response	Oscillator Frequency	Instrument Response
(Mhz)	(Volts)	(Mhz)	(Volts)
	0		······································
.2	.0022	23.	.590
. 4	.0024		
.6	.0026	24.	.69
. 8	.0027	ı	
1.0	.003	25.	.60
2.	.0045		
3.	.0071	26.	.48
4.0	.0117	27.	.385
	(28.	.325
		29.	.28
		30.	.258
		31.	.238
,		32.	.22
5.	.0155	33.	.207
.6.	.0178	34.	.195
7.	.0192	35.	.19
		36.	.18
8.	.0255		.16
		38.	.15
9.	.0243	39.	.145
		40.	.14
10.	.0297	41.	.125
11.	.04:0	42.	.12
12.	058	43.	.11
13.	.056	44.	.095
14.	.062	45.	.065
·15.	.072	46.	.055
16.	.088	47.	.048
17.	.098	. 48.	.045
• • • • • • • • • • • • • • • • • • • •	7	49.	.045
18.	.132	50.	.045
19.	.168	7	
20.	.220		
21.	.305	· ·	
22.	.420		

2

Sample Number

Sample Composition (percent by weight)

Water - 2.6 Clay - 9.50 Seacoal - 5.00

Density 120 grams/cc

Oscillator	Instrument	Oscillator	Instrument
requency	Response	Frequency	Response
(Mhz)	(Volts)	(Mhz)	(Volts)
0.2	0.0025.	34.	.180
0.4	0.004 •	35.	.160
0.6	0.00475	36.	.145
0.8	0.0055	37.	.125
1.0	0.006	38.	.120
2.0	0.0085	39.	.115
3.0	0.012	40.	.105
4.0	0.016	41.	.100
5.0 4	0.02175	42.	.090 ~
. 6.	0.022	43.	0.0875
7.	0.027	44.	0.080
8.	0.0335	45.	0.080
9.	0.032	46.	0.0775
10.	0.0385	47.	0.070
Il.	0.0500	48.	0.060
12.	0.067	49.	0.0375
13.	0.068	50.	0.020
14.	0.073		
15.	0.084		
16.	0.097		· · · · · · · · · · · · · · · · · · ·
17.	0.112		
18.	0.143		
19.	0.180		
20.	0.225		·····
21.	0.300		· · · · · · · · · · · · · · · · · · ·
22.	0.420		
23.	0.480		· · · · · · · · · · · · · · · · · · ·
24.	0.72		<u> </u>
. 25.	0.66		
26.	0.52		<u> </u>
27.	0.42		
28.	0.35		
29.	0.30		· · · · · · · · · · · · · · · · · · ·
30.	.0.26		
31.	0.23.		
32.	0.215		
33.	0.195		

Water - 2.7 Clay - 9.50 Seacoal - 5.00

Density 120 grams/cc

Ambient Temperature - 70° F

Oscillator Frequency (Mhz)	Instrument Response (Volts)	Oscillator Frequency (Mhz)	Instrument Response (Volts)
0.2	0.004	34.	.180
0.4	0.00475	35.	.165
0.6	.00525	36.	.150
0.8	.0055	37.	.140
	.006	38.	.125
2.	.00825	' 39.	.115
3.	.012	40.	.105
4.	.01575	41.	.100
5.	.02175	42.	.095
6.		43.	.090
7.	.0265	44.	.085
8.	.0335	45.	.080
9.	.0320	46.	.080
10.	.0385	47.	.0775
11.	.050	48.	.070
12.	.066	49.	.050
13.	.068	50.	.025
14.	.073		
15.	.084		
16.	.090	-	
17.	.110		
18.	.145		
19.	.180		
20.	,230	,	·
21.	.305		
22.	.420		, , , , , , , , , , , , , , , , , , ,
23.	590		
24.	.720		
25.	.67		
26.	.52		
27.	.43	,	,
28.	,345		
29.	.30		
30.	. 265		
31:	.240		
32.	.215		·
33.	.195		

Sample Numbers

4, 12, 20, 28

Sample Composition (percent by weight)

Water - 2.8 Clay - 9.5 Seacoal - 5.0

Density 120 grams/cc

Ambient Temperature - 70° F

Oscillator	Instrument '	Oscillator	Instrument
Frequency	Response	Frequency	Response
(Mhz)	(Volts)	(Mhz)	(Volts)
. 2	.0040	23.	.55
.4	.0051		
.6	.0056	24.	.718
.8	.0061		·
1.0	.0066	25.	.63
2.0 3.0	.0091		
3.0	.013	26.	.52
4.0	.017	27.	.42
•		28,	.35
	•	29.	.30
	,	30.	.26
	1	31.	.23
		32.	.21
5.0	.023	33.	.19
6.0	.023	34.	.18
7.0	.028	35.	.16
	,	36.	.15
8.0	.035	37.	.135
		38.	.12.
9.0	.034	39.	.116
		40.	.107
10.0	.040	41.	.102
11.0	.051	42.	096
12.0	.068	43.	
13.0	.069	44.	
14.0	.074	45.	.065
15.0	.085	46.	arte i made e se de serre i made un i i i i i i i i i i i i i i i i i i
16.0	.098	47.	
17.0	.112	.48.	
		49.	· · · · · · · · · · · · · · · · · · ·
18.0	.147	50.	
19.0	.181		
20.0	.23		
21.0	.30	• ••••••••••••••••••••••••••••••••••••	
22.0	.41		

. 5

Sample Number

Sample Composition (percent by weight)

Water - 2.9 Clay - 9.50 Seacoal - 5.00

Density 120 grams/cc

<u> </u>			•
Oscillator	Instrument	Oscillator	Instrument
Frequency	Response	Frequency	Response
(Mhz)	. (Volts)	(Mhz)	(Volts)
0.2	.004	. 34.	.175
0.4	.0055	35.	.160
0.6	.006	36.	.150
0.8	0067	37.	.133
1.0	.0075	38.	.120
2.	.0105	39.	.115
3.	.0145	40.	.108
4.	.0195	41.	.103
5.	.025	42.	.10
6.	.0255	43.	.09
7.	.030	44.	.085
8.	.0385	45.	.07
. 9.	.036	46.	.05
10.	.0425	47.	045
11.	.054	48.	.04
12.	.068	49.	.035
13.	.072	50.	.035
14.	.076		.000
15.	.088		
16.	.104		
17.	.114		
18.	.150		
19.	.185		
20.	.230		
21.	.300		*
$\frac{22}{22}$.	.415		•
23.	.560	•	
24.	.710		
25.	.67		
26.	.52		,
27.	.42		
28.	.35		
29.	.30		-
30.	.26		
31.	.23		
32.	.21		
33.	.19		1

Water - 3.0 Clay - 9.50 Seacoal - 5.0

Density 120 grams/cc

Oscillator	Instrument	Oscillator	Instrument
Frequency	Response	Frequency	Response
(Mhz)	(Volts)	(Mhz)	(Volts)
. 2	009	23.	.580
. 4	.0095		
. 6	.0098	24.	.72
. 8	.010	•	
1.0	.0102	25.	.68
2.0	.0123		
3.0	.0169	26.	.53
4.0	0208	27.	.42
-		28.	. 34
1		29.	. 29
		30.	.252
		31.	.225
		32.	.200
5.0 .	.027	33.	.180
6.0	.027	34.	.164
7.0	.032	35.	.150
•	• 1	36.	.137
8.0	.041	37.	.122
	P	38.	.111
9.0		39.	.105
•		40.	.098
10.0	.0423	41.	.092
11.0	.0565	42.	.089
12.0	.068	43.	.085
13.0	.072	44.	.080
14.0	.078	45.	.065
15.0	.089	46.	. 045
16.0~	.104	47.	.037
17.0	.115	48.	.033
		49	.031
18.0	.150	50.	.035
19.0	.188		
20.0	.234		
21.0	.305	The second section of the section	***********
22.0	.420		•

Water - 4.0 Clay - 9.5 Seacoal - 5.0

Density 120 grams/cc ·

Ambient Temperature - 72^OF

Oscillator	Instrument	Oscillator	Instrument
Frequency	Response	Frequency	Response
(Mhz)	(Volts)	(Mhz)	(Volts)
		. 22.	.43
. 2	.010		
. 4	.0105	23.	.60
.6	.0108		
. 8	.011	24.	.76
1.	.0117		
2.	0148	25.	.72
3.	020	,	
4.	.025	26.	.56
	1	27.	.44
		28.	.35
•		29.	•30·
4.7		30.	.25
		31.	.22
M-		32.	.19
5.	.032	33.	.17
6.	.032	34.	.155
7.	.038	35.	. 14 .
		36.	.125
8.	.048	37.	, .115
		38.	.10
9.	.043	39.	.095
	,	40.	.09
10.	.051	41.	.086
11.	.064	42.	.085
12.	.073	43.	.08
13.	.079	44.	.07
14.	.085	45.	.047
15.	.098	16.	.032
16.	.113	47.	.03.
17.	.130	48.	.025
		49.	.027
18.	.160	50.	.03
19.	.195	·	- total and a second se
20.	.248		
21.	.320		

8

Sample Composition (percent by weight)

Water - 5.0 Clay - 9.5 Seacoal - 5.0

Density 120 grams/cc

Ambient Temperature - 70° F

Oscillator Frequency (Mhz)	Instrument Response (Volts)	Oscillator Frequency (Mhz)	Instrument Response (Volts)
.2	0157	23.	.62
. 4	.0167		
.6	.017	24.	.80
. 8	.0175		. ,
1.	.0178	25.	78
2.	.021		
3.	.0266	26.	.60
4.	.032	27.	.46
•	•	28.	.37
		• 29.	.30
· · · · · · · · · · · · · · · · · · ·	• • • • • • • • • • • • • • • • • • • •	30.	.26
		.31.	22
		32.	.195
5.	.040	33.	· .172
. 6.	.0385	34.	.152
7.	.0445	35.	.139
		36.	.122
8.	.058	37.	.11
•		38.	.10
9,	.051	39.	.095
		40.	.09
10.	.059	41.	.085
11.	.072	42.	.08
12.	.085	43.	.045
13.	.087	44.	.032
14.	.094	45.	.032
15.	.106	46.	.036 .
16. 4.	.130	47.	.037
17.	.143	48.	.035
		49.	∘.034
18.	.168	50.	.035
19.	.210		
20.	.258		
	.335		
21.	1 .335 1		

Water - 2.8 Clay - 9.5 Seacoal - 4.00

Density 120 grams/cc

Ambient Temperature - $72^{\circ}F$

Oscillator	Instrument	Oscillator	Instrument
Frequency	Response	Frequency	Response
(Mhz)	(Volts)	(Mhz)	(Volts)
. 2	.0062	23.	.565
. 4	.0066		
.6	.0068	24.	.700
. 8	.007		
1.0	.0072	25.	.640
2.0	.009		
3.0 .	.013	26.	.500
4,0	.0163	27.	.397
		28.	.325
		29、	280
		30.	.245
		31.	220
,		32.	.200
5.0	.022	33.	182
6.0	.022	34.	.168
7.0	.0268	35.	.154
		36.	.140
8.0	.034	37.	.129
		38.	.118
9.0	.032	. 39.	:110
		40.	.102
10.0	.038	41.	.096
11.0	.049	42.	.091
12.0	.064	43.	.085
13.0	.065	• 44.	.068
14.0	.071	45.	.043
15.0	.081	46.	.038
16.0	.096	47.	.040
17.0	.107	48.	.038
<u> </u>		49.	.037
18.0	.140	50.	.038
19.0	.175		
20.0 .	.210	, , ,	
.21.0	.300		
22.0	.410		1
	1		

Sample Number

Sample Composition (percent by weight)

Water - 2.8 Clay - 9.50 Seacoal - 4.70

Density 120 grams/cc

Oscillator . Frequency (Mhz)	Instrument Response (Volts)	Oscillator Frequency (Mhz)	Instrument Response (Volts)
. 2	.007	23.	.57
.4	.007/3		
.6	.007	24.	.70
.8	.008		
1.	.0083	25.	.66
2.	.010	i	
3	.014	26.	.52
4.	.018	27.	.405
		28.	.34
*	1	29.	.285
		30:	.25
		·31.	.22
.		32.	.20
5.	.024	33.	.18
6.	.024	34.	.175
7.	.029	35.	.155
		36.	.145
8.	.037	37.	.135
		38.	.12
9.	.034	39.	.115.
		40.	.11
10.	.041	41.	.105
11.	.052	42.	.10:,
12.	.066	43.	.095
13.	.068	44.	.09
14.	.074	45.	.08
15.	.086	46.	.055
16.	.100	47.	.045
17.	.112	48.	.045
		49.	.035
18.	.145	50.	.035
19.	.180	J	.000
20.			
21.		An an Arresta similarinasiny silippi, diri basil apartiti ungumangay on genebagaili.	, ,
22.	.300		
44.	.415		-

Sample Number

Sample Composition (percent by weight)

Water - 2.80 Clay - 9.50 Seacoal - 4.85

Density 120 grams/cc

Ambient Temperature - $72^{\circ}F$

Oscillator Frequency	Instrument Response	Oscillator Frequency	Instrument Response
(Mhz)	(Volts)	(Mhz)	(Volts)
. 2	.007	23.	.58
. 4	.0075		
.6	.008	24.	.71
.8	.0082		
1.	.0085	25.	.66
2.	.011		
3.	0152	26.	.52
4.	.0193	27.	.41
	•	28	.335
4.		29.	.285
		30.	.25
		31.	,22
	,	32.	.20 ,
5.	.0256	33.	.18
6.	.0255	34.	.165
7.	.0305	35)	15
		36.	.14
8.	.039	37.	.13
2-		38.	.118
9.	.036	39.	.11
		40.	.105
10.	.043	41.	.10
11.	.0545	42.	.095
12.	.067	43.	.09
13	.071	44.	.085
14.	.076	45.	.075,
15.	.088	46.	.055
16.	.103	47.	.045
17.	.113	48.	.037
		49.	.03
18.	.150	50.	.03
19.	.185		
20.	.235		<u> </u>
21.	300	The street of th	
22.	,415		

Water - 2.8 Clay - 9.50 Seacoal - 5.15

Density 120 grams/cc

Ambient Temperature - $72^{\circ}F$

Oscillator Frequency	Instrument Response	Oscillator Frequency	Instrument Response
(Mhz)	(Volts)	(Mhz)	(Volts)
.,2	.0052	23.	.580
.4 -	\$0057		
.6	. 006	24.	.720
· .8	.006		
<u> </u>	.0062	25.	.65
2.	.008	*	1
ું3.	.012	26.	.51
4	.016	27.	.41
	_	28.	. 34
		29.	.295
,	1	30.	.255
1	, 1	31.	.230
		32.	.210
5.	.0218	33.	.195
6.	.022	34.	.180
7.	.0265	35.	.165
		36.	.150
8	.0335	37.	.140
*		38.	.127
9.	.032	39.	.120
		40.	.11
10.	.049	41.	.105
11.	.050	42.	.09
12.	.065	43.	.06
13.	.065	44.	.045
14.	.072	45.	.047
_15.	.083	46.	.05
16.	.098	47	.047
17.	.108	.48.	.045
		49.	.045
18.	145	50:	.045
19.	.180	, ,	
20.	.230		*
21.	:305		
22.	.420	<u>'</u>	· · · · · · ·

Water - 2.8 Clay - 9.50 Seacoal - 5.30

Density 120 grams/cc
Ambient Temperature - 72°F

Oscillator	Instrument	Oscillator	Instrument
Frequency	Response	Frequency	Response
(Mhz)	(Volts)	(Mhz)	(Volts)
. 2	.0065	23.	.58 ,
.4	.007		*
.6	.0073	24.	.72
.8	.0078		
1.	.008	25.	.66
2.	.0103		
3.	.0145	26.	.52
4.	.0185	27.	.42
		28.	.34
		29.	. 29,5
		30.	. 255
		31.	.223
		32.	.202
5.	.0247	33.	.190
6.	.025	34.	.170
7.	.0295	35.	.157
		36.	.145
8.	.038	37.	.130
		38.	.120
9.	.035	39.	.112
	1	40.	.107
10.	.042	41.	.104
li.	.0535	42.	.095
12.	.066	43.	.067 -
13.	.070	44.	.041
14.	.076	45.	.041
15.	.087	46.	.043
16.	.102	47.	. 045
17.	.112	48.	.044
all F & .		, 49.	.045
18.	.147	50.	.045
19.	.180		
20.	.230		• ,
21.	.305		
22.	.42		
***	1		

Water - 2.8 Clay - 9.5 Seacoal - 6.0

Density 120 grams/cc

Oscillator Frequency (Mhz)	Instrument Response (Volts)	Oscillator Frequency (Mhz)	Instrument Response (Volts)
. 2	.0067	23,	.580
. 4	.007		
.6	.0072	24.	.715
.8	.0075	, ,	
1.0	.0076	25.	.670
2.0	.0094		
3.0	.0135	26.	520
4.0	.0172	27.	.410
		28.	.340
		29.	,290
		30.	.255
	· · · · · · · · · · · · · · · · · · ·	31.	.225
		32.	.205
5.0	.023	33.	.190
6.0	.0231	· 34.	.172
7.0	.028	35.	.160
	1	36.	.145
8.0	.036	37.	.132
		38.	.120
9.0	.033	39.	.112
		40.	.105
10.0	.040	41.	.097
11.0	.0515	42.	.079
12.0	.065	43.	.051
13.0	.068	44.	.050
14.0	.073	45.	.050
15.0	.084	46.	051
16.0	.099	47.	.050
17.0	.110	48.	.049
		49.	.048
18.90	.145	50. 4	.048
19.0	.180	,	
20.0	.228		
21.0	.310	•	· •
22.0	.410		

Sample Number

16

Sample Composition (percent by weight)

Water - 2.8 Clay - 9.5 Seacoal - 7.0

Density 120 grams/cc

	· · · · · · · · · · · · · · · · · · ·		
Oscillator	Instrument	Oscillator	Instrument
Frequency	Response	Frequency	Response
(Mhz)	(Volts)	(Mhz)	(Volts)
. 2	.005	23.	.595
.4	.0052		
.6	.0053	24.	.720
6.8	.0056		, '
1.0	.0059	25.	.645
2.0	.0077	1	
3.0	.0112	26.	.520
4.0	.015	27	.410
		28.	.340
^		29.	.295
,	·	30.	.260
		31.	.240
		32.	.215
5.0	.0208	33. 34.	.198
6.0	.0208	34.	.183
7.0	.0255	35.	.169
7.5	.029	36.	.155
8.0	.031	37.	.140
8.5	.030	38.	.130
. 9.0	.0305	39.	. 122
9.5	.0335	40.	.113
10.0	.037	41	.104
11.0	048	42.	.082
12.0	.065	4\3.	.055
13.0	.067	44.	.050
14.0	.0675	45.	.052
15.0	.082	46	:051
→ 1.6.0	\ .097⋅	47.	.050
17.0	.109	48.	.049
300		49.	.048
18.0	.142	50.	.048
19.0	.177	•	
20.0:	.230		
21.0	.310		
22.0	. 420		

17

Sample Composition (percent by weight)

Water - 2.8 Clay - 8.0 Seacoal - 5.0

Density 120 grams/cc

		1	T
Oscillator .	Instrument	Oscillator	Instrument
Frequency	Response	Frequency	Response
(Mhz)	. (Volts)	(Mhz)	(Volts)
	1006		
2	.006		ļ
. 4	.0067	23.	.580
.6	.007		
. 8	.0071	24.	.70
1.	.0075	<u> </u>	<u> </u>
2	.0095	25.	.64
3.	.0135		
4.	.0174	26.	.505
	٠	27.	.400
		28.	335
		29.	.28
		30.	.25
		31.	.22
5.	,0235	32.	.20
б.	.0232	33.	.185
7.	.028	34.	.17
		35.	.16
8.	.036	36.	.142
		37.	.130
9.	.033	38.	.120
	· · · · · · · · · · · · · · · · · · ·	39.	.113
10.	.040	40.	.108
11.	.0515	41.	.100.
12.	.064		.090
13.	.068	43.	.055
14.	.072	44.	.042
15.	.084	45.	,045
16.	.097	46.	.046
17.	.108	47.	.047
11.	•100	48.	.046
10 0	1 1 1 1		
18.0	.145	49.	.045
19.	.180	50.	.045
20.	.226		
$\frac{20.}{21.}$			
	.300	<u> </u>	• •
22.	. 415	L	

Sample Number

Sample Composition (percent by weight)

Water - 2.8 Clay - 9.00 Seacoal - 5.00

Density 120 grams/cc

Ambient Temperature - 72°F

Oscillator Frequency	Instrument Response	Oscillator Frequency	Instrument Response
(Mhz)	(Volts)	(Mhz)	(Volts)
(11112)	(VOILS)	(11112)	(VOICS)
0.2	.0068	34.	.17
0.4	.007	35.	.157_
0.6	.0075	36.	.145
0.8	.0078	37.	.13
1.0	.008	38.	.12
2.0	.010	39.	.11
3	.014	40.	.10
4.	.018	41.	08
5.	.0245	42.	.05
6. ·	.0243	43.	.05
7	. 029	44.	.05
8	.0375	45.	.05
9	.0345	46.	.05
10.	.042	47.	.05
11.	.053	48.	.05
12.	.066	49.	.05
13.	.070	50.	.045
14.	.075.		
15.	.086		•
16.	.100		
17.	.112	*	·
18.	.147	,	- \
19.	.180		
20.	230		
21.	.305		
22.	.420		
23.	.580		,
24.	.720		
25.	66		
26.	57		
27.	.415		
27. 28.	. 34	. /	
29.	29		
30.	.415 .34 .29 .255 .225 .205		
31.	225		
31.	205		
33.	10		

Water - 2.8 Clay - 9.25 Seacoal - 5.00

Density 120 grams/cc

Ambient Temperature - 70°F

Òscillator	Instrument	Oscillator	Instrument
Frequency	Response	Frequency	Response
(Mhz)	(Volts)	(Mhz)	(Volts)
			
0.2	.005	34.	.163
0.4	.006	35.	.158
0.6	.0065	36.	.14
0.8	.007	37.	.125
1.0	.0075	38.	.115
2. 3.	.010	. 39.	.11
3	014	40.	.10
4.	.018	41.	.072
5.	.024	42.	. 044
6.	.0245	43.	.026
· 7.	.0295	44.	.028
8.	.037	45.	.029
9.	.035	46.	.029
. 10.	.042	47.	.028
11.	.053	48.	.026
12.	.067	49.	.024
13	.07 . *	50.	.0235
14.	.076		•
15.	.086	*	
16.	.101		,
17.	.112		
. 18,	.148		
19.	.185		· · · · · · · · · · · · · · · · · · ·
20.	.230		
21.	.305		•
22,	.420	•	
23.	.580		•
24.	.72		
25.	.67		
26.	.52		•
27.	. 41		•
28.	.338		•
28. 29.	.338 .284 .25 .22 .20		-
30. ·	.25	,	·
31.	.27		· · · · · · · · · · · · · · · · · · ·
31. 32. 33.	20		
33.	10		

Sample Number

Sample Composition (percent by weight)

Water - 2.8 Clay - 9.75 Seacoal - 5.00

Density 120 grams/cc

Ambient Temperature - 71° F

Oscillator Frequency	Instrument Response	Oscillator Frequency	Instrument Response
(Mhz)	(Volts)	(Mhz)	(Volts)
.2	.005	23.	.58
. 4	.0058		
. 6	.006	24.	.72
. 8	.0064		
1.	.0068	25.	.67
2.	.0088		
3.	.0128	26.	.52
4.	.0215	27.	.41
		28.	. 34
	• ,	29.	.295
		30	26
		31.	
•		32.	21
5.0	.0225	33.	.19
6.	.0225	34.	.175
7.	.0275	35.	.16
		36.	.147
8.	035	37.	.133
		38.	.12
9.	.033	39.	.113
· · · · · · · · · · · · · · · · · · ·		40.	.105
10.	.04	41.	.10
11.	.051	42.	.08
12.	.066	43.	.05
13.	.068	44.	.05
14.	,074.	45.	05
15.	.085	46.	.05
16.	.100	47.	.05
<u> </u>	.11	48.	.05
		49.	. 05
18.	.147	50.	.045
19.	.190		
20.	.230 /		
21.	.305		
22.	.42	<u> </u>	

Water - 2.8 Clay - 10.00 Seacoal - 5.00

Density 120 grams/cc

Ambient Temperature - 71°F

Oscillator	Instrument Response	Oscillator Frequency	Instrument Response
(Mhz)	(Volts)	(Mhz)	(Wolts)
. 2	.005	23.	.575
, 4	.0055		
.6	.006	24.	.71
8	.0062		
1.	.0068	25.	.66
2.	.009		
3.	013	26.	.52
4.	.017	. 27.	.40
	* *(1)	28. 6	.34 .
	-	29.	28
		30.	.25
		31.	.22
		32.	.205
5.	.0226	33.	.195
6	023	34.	.18
7.	.028	35.	16
	```	36	.15
8	.035 ,	. 37.	.135
•	·	38.	.125
9	.033	39.	.12
		40.	.11
10.	.040	41.	.105
ll:	.0515	42.	:10
12.	.07	43.	.095
13.	.068	44.	.08
14.	.075	45	.045
15.	.084	46	. 04
16.	.100	∠ 47.·	.04
17.	.110	48.	. 04
	· · · · · · · · · · · · · · · · · · ·	49	.04
18.	.145	50.	.04
19.	.180		
20.	.230		•
21.	.30	i	,
22.	.41.		·

Water - 2.8 Clay - 11.0 Seacoal - 5.0

Density 120 grams/cc

Ambient Temperature - 72°F

Oscillator	Instrument	Oscillator	Instrument
Prequency	Response	Frequency	Response
(Mhz)	(Volts)	(Mhz)	(Volts)
.2	.0042	23.	. 585
. 4	.0048		
.6	.0052	24.	.71
. 8 .	.0058	,	
1.0	.006	25.	.64
2.0	.008		, ,
3.0	.0116	26.	. 5·0
4.0	.0152	27.	.40
		28.	.335
,		29. 30.	.285
		30.	.26
		31.	.23
		32.	.21
5.0	.021	33.	<u> 195</u>
6.0	021	34.	.180.
7.0	.0255	35.	.167
		36.	.152
8.0	.032	·37.	.140
	, ,	38.	.128
9.0	.031	39.	.120
	,	40.	.110
10.0	.0375	41.	.105
11.0	.048	42.	.095
12.0	.064	43.	.060
13.0	.065	. 44.	.048
14.0	.070	45.	.050
15.0	.081	46.	.050
16.0	.100	47.	.050
17.0	107	48.	.048
		~49.	.045
18.0	.140	.50.	.047
19.0	177		
20.0	.226		
21.0	.305		
22.0	.420		<u> </u>

Sample Number

Sample Composition (percent by weight)

Water - 2.8 Clay - 12.0 Seacoal - 5.0

Density 120 grams/cc

Ambient Temperature - 72°F

Oscillator Frequency	Instrument Response	Oscillator Frequency	Instrument Response
(Mhz)	(Volts).	(Mhz)	(Volts)
.2	006	23.	.580
. 4	.0062		
.6	.0065	24.	.710
. 8	.0067		
1.0	.007	25.	.650
2.0	.0087		
3.0	.0126	26	.510
4.0	0162	27.	.408
		28./	.340
		. 29.	.290
•		30.	.255
	·	31.	.23
		32.	.206
5.0	.022 .	33.	.190
6.0	.0218	34:	172
7.0	.0265	35.	.158
		36.	.145
8.0	.034	37.	.130
		38.	.118
9.0	.032	39.	.110
*		40.	.102
10.0	.038	41.	.098
11.0	.049	42.	.092
12.0	.064	43.	.090
13.0	.066	44.	.080
14.0	.072	45. ;	.058
15.0	.083	46.	· .038·
16.0	.098	47.	.037
17.0	.110	48.	.034
		49.	035
18.0	145	50.	.038
19.0 ~	.180		•
20.0	226		
21.0	.300		•
22:0 .	.407		<u> </u>

# Sample Number

Sample Composition (percent by weight)

Water - 2.8 Clay - 9.50 Seacoal - 5.00

Density 100 grams/cc Ambient Temperature - 72.8°F

Oscillator Frequency	Instrument Response	Oscillator Frequency	Instrument Response
(Mhz)	(Volts)	(Mhz)	(Volts)
		25.	.59
, 2	.0025		
. 4	\ .003	26.	.46
.6	.003	. 27.	.37
. 8	.0032	. 28.	.31
1.0	.0037	29.	.27
2.	.0055	30.	.24
3.	,0081	31.	
4.	0115	32.	.205
		33.	.19
		34.	.18
		.35,	.17
	·	36.	.16
		37.	.15
5.	.0165	38.	.14
6.	.018	39.	.13
7.	.0205	. 40.	12
8.	.0255	41.	.10
9	.025	42.	.06
10.	.030	43.	.06
11.	.04	. 44.	.06
12.	.0565	45.	.06
13.	.0555	46.	.06
14.	.0595	47.	, .06
15.	.070	48.	058
16	.084	49.	055
17.	.094	50.	.052
18.	.116		
19.	.160		
20	.21	\	
<b>21.</b> c	28	\	
22.	.395		
23.	.545	,	1
24.	.66		

Water - 2.8 Clay - 9.50 Seacoal - 5.00

Density 110 grams/100 cm³
Ambient Temperature - 72°F

<b>,</b>			
Oscillator	Instrument	Oscillator	Instrument
Frequency	Response	Frequency	Response
(Mhz)	· (Volts)	(Mhz)	(Volts)
	·	32.	.210
. 2	.00175	33.	:190
, .4	.003	34.	175
.6	.0035	35.	.160
.8	.0044	36	.150
1.	.005	37.	140
2,	008	38.	125
3.	.011	39.	.120
. 4.	.015	40.	.110
		41.	.105
5.	.020	42.	100
6.	.021	43.	.090
7.	.025	44.	.080
8.	.031	45.	
9.	.030	. 46.	.040
10.	.036	47.	.040
11.	.0475	48.	.040
12.	.064	49.	.040
13.	.064	50.	040
14.	.069	· /.	
15.	.080		,
16."	.094		, ~ ~
17.	.104		
18.	.140		•
19.	.175		
20.	. 220	<u>-</u> ,	
21.	.290		
22.	.400		
23.	.560		
24.	. 690		
25.	.640		
26.	.500	الله الله الله الله الله الله الله الله	
27.	.400	,,	
28.	335	,	
29.	.335		
30.	.250		1.
31.	.230		

Water - 2.8 Clay - 9.50 Seacoal - 5.00

Density 115 grams/cc

Ambient Temperature -  $72^{\circ}F$ 

Oscillator Frequency	Instrument Response	Oscillator Frequency	Instrument Response
(Mhz)	(Volts)	(Mhz)	(Volts)
•		32.	.21
.2	.004	33.	.20
.4	.00525	34.	.17
.6	.0055	. 35.	.16
. 8	.006	36.	.15
1.	.00675	37.	.135
2.	.009	38.	.12
3.	.013	39.	11
4.	.017	40.	105
		41.	~ · · · · · ·
5.	.0165	42.	
6.	.023	43.	
7.	.0275	44.	
8.:	.035	45.	.05
9.	.033	46.	, .
10.	.040	47.	
11.	.051	48.	,
12.	.066	49.	
13.	.068	50.	,02
14.	.072		
15. ·	. 084		
-16. ·	.098		
17.	.110		
18.	.145		r
19.	.180		
20.	.225		
_21	.295		
22.	.410		
23.	.565	•	
24.	.700	•	
25.	.660		
26.	.520		
27.	.420		
28.	.340		
29.	.30	•	1
30.	.26		, ,
31.	.23		

Water - 2.8 Clay - 9.50 Seacoal - 5.00

Density 125 grams/cc

Ambient Temperature - 72°F

,			
Oscillator	. Instrument	Oscillator	Instrument
Frequency	Response	Frequency	Response
(Mhz)	(Volts)	(Mhz)	(Volts)
		33.	.20
. 2	.005	34.	.18
.4	.0055	35.	.17
.6	.006	36.	.15
.8	.00625	. 37.	.14
l.	.0065	38.	.127
2.	.0085	39.	.12
3.	.0125	40.	.11
4.	.0165	41.	.105
5.	.022	42.	.10
6.	.0225	43.	.09
7.	.0272	44.	.078
8.	.034	45.	.050
9.	.033	46.	.040
10.	.039	47.	.040
11.	.050	48.	.040
12.	. 066	49.	040
13.	.068	50.	.040
14.	.074	4.6	.024
15.	.084		*
16.	1.100		
17.	.112	1	
18	.150		
19	.185		, ,
20.	.230		
.21.	.305		
22.	.41		
23.	.58		
24.	.72		
25.	.66		
26.	.52		4
27.	.42		
28.	.36	TO THE REAL WORLD WIND WIND WIND WIND WIND WIND WIND WIN	
29.	.30		· · · · · · · · · · · · · · · · · · ·
30.	26		· · · · · · · · · · · · · · · · · · ·
31.	.24		,
32.	.21	· · · · · · · · · · · · · · · · · · ·	
		<del></del>	

Water - 2.8 Clay - 9.50 Seacoal - 5.00

Density 130 grams/cc

Ambient Temperature -  $72^{\circ}F$ 

Oscillator	Instrument	Oscillator	Instrument
Frequency	Response	Frequency	Response
(Mhz)	(Volts)	(Mhz)	(Volts)
	•	32.	.22
.2	.005	33.	.20
.4	.006	34.	.18
.6	.006	35.	.165
.8	.007	36.	.150
1.	.0075	37.	.140
2.	.010	38.	.125
3.	.014	39.	.120
4.	.0185	40.	.11
		41.	.105
5.	.0245	42.	,10
6.	.025	43.	.095
7.	.030	44.	.085
8.	.038	45.	.060
9.	.036	46.	.045
10.	.043	47.	.040
$\frac{10}{11}$ .	.055	48.	.040
12.	.069	49.	.040
13.	.073	50.	.040
14.	.078		
15.	.090		
16.	.106		
17.	.116		
18.	.155		
19.	.190		<del></del>
20.	. 240		
21.	.310		
22.	.425		· · · · · · · · · · · · · · · · · · ·
23.	.587	·	
24.	.740		
25.	.680		
26.	.540		· · · · · · · · · · · · · · · · · · ·
27.	.440		
- 28.	.360		· · ·
7 29.	.31		
30.	.27		•
31.	.24	mangina managan an ang ang ang ang ang ang ang an	

Water - 2.8 Clay - 9.50 Seacoal - 5.00

Density 140 grams/cc Ambient Temperature - 72°F

Oscillator	Instrument	Oscillator	Instrument
Frequency	Response	Frequency	Response
(Mhz) *	(Volts)	(Mhz)	(Volts)
,		25.0	.750
. 2	.011		`
. 4	.012	26.0	.600
.6	.0123	27.	.460
.8	.0125	28.	37
1.0	.013	2,9.	.31
2.	.0157	30.	.26
3.	.021	31.	.23
4.	026	32.	.20
		33,	.18
		34.	.16_
		35.	.15
,	1	36.,	.135
,		37.	.12
5.0	.0335	38.	.11
6.0	.033	39.	.105
7.0	.038	40.	.10
8.0	.050	41	. 08
9.0	.044	42.	.05
10.0	, .052	43.	.04
11.	.072	44.	.04
12.	.076	45.	.045
13.	. \ .082	46.	.045
14.	.088	47.	.045
15.	.100	48.	. 045
16:	.115	49.	.040
17.	.138	50.	.040
18.	165	1	,
19.	.200		
20.	.250		
21.	.310		
22. ·	.435		•
23.0	.600		
24.0	.760	•	

5

Sample Composition (percent by weight)

Water - 2.8 Clay - 9.50 Seacoal - 5.00

Density 150 grams/cç

Ambient Temperature - 72°F

Oscillator	Instrument	Oscillator	Instrument
Frequency	Response	Frequency	Response
(Mhz)	(Volts)	(Mhz)	· (Volts)
	11	32.	.210
: 2	.013	33.	.190
. 4	.014	34.	.165
.6	.014	35.	.150
. 8	.0145	36.	.140
1.0	.015	37.	• I Z U
2.	.0174	38.	.110
3.	.0226	39.	.105
4.	.0275	40.	.100
		41.	.095
5.	.035	42.	.090
6.	035	43.	.086
7,	.040	44.	.080
8.	.0525	45.	.060
9.	.046	46.	.052
10.,	.054	47.	.035
11.	.068	48.	.030
12.	.080	49.	.030_
13.	.084	50.	.030
14.	.092		
15.	.104		
16.	.120		
17.	.140		•
18.	.170		
19.	.210		
20.	.255.		
21.	.330		,
22.	.440		
23.	.600		
24.	6.780	•	
25.	.780		
26.	610		
27.	.480		
28.	400		
29.	.320		
30.	.280		e de ar ann a la la la marathaire ann ann an
31.	.240		***

O

Water - empty Clay -Seacoal -

Density'

Ambient Temperature

Oscillator Frequency (Mhz)	Instrument Response (Volts)	Oscillator Frequency (Mhz)	Instrument Response (Volts)
. 2	0	23.	.250
. 4	0		<del> </del>
.6	0	24.	.300
. 8	0		
. 1.0	0	25.	.210
2.0	.0005		
3.0	.002	26.	A 55
4.0	.0028	27.	.120
	•	28.	.100
		29.	.072
1		30.	.064
	A AN AND AND AND AND AN AN AN AN AND AN AN ANALYSIS OF THE ANA	31.	.056
		32.	.051
5.0	.0038	33.	.048
	.0055	34.	.045
6.0 7.0	.0045	35.	.044
the set of the set of the second		36.	.042
8.0	.0083	37.	.040
	· · · · · · · · · · · · · · · · · · ·	38.	039
9.0	.0069	39.	.035
***************************************		40.	.034
10.0	.007	41.	
11.0	.012	42.	<del></del>
12.0	.019	43.	
13.0	.014	44.	
14.0	.015	.45.	.036
15.0	.019	46.	
16.0	.024	47.	
17.0	.027	48.	**
	And the second s	49.	
18.0	.034	50.	.028
19.0	.044	_	<del></del>
20.0	.059		*
21.0	.090		
22.0	.155	,	etti entimente e manto una apropara en acceptada en el constitución de la constitución de la constitución de l

### Curve Fitting Using the Method of Least Squares

Assume the existence of a table of n + 1 data points  $(X_0,Y_0)$ ,  $(X_1,Y_1)$ ,  $(X_2,Y_2)$ ,  $(X_3,Y_3)$ , ....,  $(X_n,Y_n)$  and an empirical function that approximates this data set called f(X).

The difference between the empirical function f(X) at point  $(X_i, Y_i)$  and the actual curve may be written as

$$\varepsilon_i = F(X_i) - Y_i$$

The  $\epsilon i$  can be computed for each data point, squared and summed to get  $M = \sum_{i=0}^{n} \left[ f(X_i) - Y_i \right]^2$ 

If f(X) is a straight line  $f(X) = C_0 + C_1X$ 

then 
$$M = \sum_{i=0}^{n} \left[ C_0 + C_1 X_i - Y_i \right]^2$$

M can be considered as a function of  $C_0$  and  $C_1$ . A necessary condition such that f(X) is the best approximation for this data set is that  $\frac{\partial M}{\partial C_0} = \frac{\partial M}{\partial C_1} = 0$ 

hence

$$\frac{\partial M}{\partial C_0} = 2 \sum_{i=0}^{n} (C_0 + C_1 X_i - Y_i) = 0$$

$$\frac{\partial M}{\partial C_1} = 2 \sum_{i=0}^{n} (C_0 + C_1 X_i - Y_i) X_i = 0$$

$$\sum_{i=0}^{n} C_{0}X_{i} + \sum_{i=0}^{n} C_{1}X_{i}^{2} = \sum_{i=0}^{n} X_{i}Y_{i}$$

which can be simplified to obtain

$$nC_0 + C_1 \sum_{i=0}^{n} X_i = \sum_{i=0}^{n} Y_i$$

$$C_{0} \stackrel{\Sigma}{\underset{i=0}{\sum}} X_{i} + C_{1} \stackrel{\Sigma}{\underset{i=0}{\sum}} X_{i}^{2} = \stackrel{\Sigma}{\underset{i=0}{\sum}} X_{i} Y_{i}$$

This is a system of two equations in two unknowns. <

The calculations which follow relate to the data presented in tables 5-2,3,4,5,&6. A straight line approximation was obtained for each data set and displayed graphically in figures 5-6,7,8,9,&10 respectively.

### Refer to table 5-2

X = water content

Y = response

X	X	•	,	٠	FREQU	ENCY	,	,	
		,8	Mhz	1	3 Mhz	16	Mhz	2	0 Mhz
		Y	XY	Y	XY	Y	XY·	Y	ХŸ
2.00 2.60 2.70 2.80 2.90 3.00 4.00 5.00	4.00 6.76 7.29 7.84 8.41 9.00 .16.00 25.00	.0335 .0350 .0385 .0360 .0480	.0510 .0871 .0904 .0980 .1116 .1080 .1920	.069 .072 .063	.3160	.097	1	.225 .230 .230	.4850 .6210 .6440
25.0	84.30	.3080	1.0281	5620	1.8144		2.6492		5.9140

$$f(X) = C_0 + C_1 X$$

$$nC_0 + C_1\Sigma = \Sigma Y$$

$$C_0\Sigma X + C_1\Sigma X^2 = \Sigma XY$$

for 8 Mhz 
$$8C_0 + 25C_1 = .3080$$
  
 $25C_0 + 84.3C_1 = 1.0281$ 

13 Mhz 
$$8C_0 + 25C_1 = .5620$$
  
  $25C_0 + 84.3C_1 = 1.8144$ 

16 Mhz 
$$8C_0 + 25C_1 = .820$$
  
  $25C_0 + 84.3C_1 = 2.6492$ 

20 Mhz 
$$8C_0 + 25C_1 = 1.866$$
  
 $25C_0 + 84.3C_1 = 5.9140$ 

$$C_0 = 0.0053$$

$$C_0 = 0.0053$$
 $C_1 = 0.0106$ 

$$C_0 = 0.0408$$
 $C_1 = 0.0094$ 

$$C_0 = 0.0586$$
  
 $C_1 = 0.0140$ 

$$C_0 = 0.1914$$

$$C_1 = 0.0134$$

Refer to Table 5-3

×	<b>*</b> ×			• •	FREQUENCY	NCY			,
*	•		8 Mhz	13	Mhz	16	Mhz	20	0 Mhz
•	,	) <del>)</del>	XX ·	×	XX	; <b>&gt;</b>	XX	<b>X</b>	X
0	6.00		.1360	. 9	60	9	4	-	40
4.70	22,090	.037	1739	.068	.3196	001.	.4700	.232	1.0904
ω.	3.52	0	89	7	44	0	99	3	.139
٥.	5.00	3	85	9	.3450	9	90	m	.150
۲.	6.52	$\sim$	72	9	34	9	04	$\sim$	.184
٣.	8.09		.2014	~	71	0	0	$^{\circ}$	19
0	6.00	3	16	9	.4080	9	94	$^{\sim}$	.368
oʻ.	9.00	m	17	9	69	σ	79	$\sim$	.610
42.00	. 226.225	.2835	1.4910 ·	.543	2.8518	.793	4.1619	1.825	9.6017
							6		ì
<b>4</b> 4	$(X) = C_0 +$	c ₁ x	·	~ ~ ~	•		,		
			, 	` `		`\	` _		
•	. ບໍ່.		. 0280 .	6990.0	0	1004	0.2090	0	·
	ບ		0.0005	0.0002	. 1	0.0002	0.0037	7	

Refer to Table 5-4 X = Clay Content, Y = Current Response

		γ · · · · · · ·			<u>,</u>					4			r	,
	20 Mhz	XX	.80	127	.185	.242	300	86	711.	17.9310		0.02343		-0.0006
,	2	Y	. 226	n	$\sim$	$\sim$	$\sim$	2 0	V	1.828	,	0	·	-0-
	16. Mhz	XX	76	34	31	75	000	1.1000	9/T.	7.7923		0.0982	•	1,000.0
FREQUENCY	7.	<b>&gt;</b> .	760.	0	6	0	0	0	ות	.794		0.0		0.0
FRI	3 Mhz	ХX	.5440.	47	55	63	80	15	76	5.3270	\(\frac{1}{2}\)	0.0782		-0.0010
	H	7	.068	1.	9	9	9	9	ا 0	. 544		0.	:	0
	Mhz	XX.	. 2880	3423	.3325	.3413	.3500	.3520	.4030	2,7515	<b>,</b> (.	.0451	,	0.0010
•,	8	Ж	.036	37	സ	က	ᠬ	$\omega$	າ ]	.2815	·•	0		
X X			64.000	7.50 5.50	0.25	5.06	00.00	1.00	44.00	780.875	· .	0,0		C ₁
×	,	• •	8.00	S	ĽΩ	7.	0.0	0	2.0	78.50				

Refer to Table 5-5

X = Density, Y = Response

		,									
	.zuw	XX	10	58 76	87	.3825	2,3187		0.1180	.,	0.0925
!	20	Ť.	10	0,W	<b>6</b> 4	.250	1.860		0		0.
4	Mhz	XX	.0840.	12 17	25	-0	1.0215		0,0147	•	0.0704
ENCY	. 16	×	.084 .094	, Φ Φ,	00	.115	.815		0,4		0.0
FREQUENCY	3 Mhz	. XX	.0565	ω N	4	44	.7042		0.0242	•	Q.0374
	٦	×	.0555	ဖ ့ဖ	9	$\infty \infty$	.5635		0.0		0.0
	Mhz	ХX	25 34	40 42	42	. 0700	.3825		0.0298		0.0545
	ω	. x	.0255	35	34 38	.0500	3010		0.0-	•	0.0
c	*×		1.0000	.440	.562	.250	12.4350	n'	. 0		į et
	×		1.00	۲.	4. 6.	4. S	9.90	~	၁	,	

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Refer to Table 5-6

E Current Response × = Total weight of water between plates (grams)

f.(x) =	$c_0 + c_1 x$				FREQUENCY	ENCY		•	
<u>,</u> ×	×	8	Mhz	1	W W	7	6 Mhz	20	Mhz
	,	.₩.	ХX	¥	λX	X	XX	X	XX
4.	.760	25	19	56	34	$+\infty$	11	100	28
3.08 3.08	9.4864	.0255	0955	.0555	.1554	.0.84	.2352	220	. 5880
۳.	9.734	33	04	68	12	9	02	2	0.2
.7	0.368	35	12	68	19	σ,	15	2	24
2.	0.497	33	08	89	20	<b>\$</b>	91	m	45
۵.4	787.	η γ γ	7 T	るなって	7 6	y C	א ה א ר	<b>7)</b> (1)	70
. 2	2.250	34	19	89	3 8	$\circ$	50	m	0.5
•	2.960	36	29	63	26	0	9	2	10
φ.	3.249	38	38	73	65	0	85	マ	73
6	5.366	30	96	82	21	$\boldsymbol{\vdash}$	50	S	80
7	7.640	52	20	84	52	2	04	S	.07I
ω.	3.040	48	30	79	19	-1	42	4	90
o.	6.000	53	48	87	22	$\sim$	80	ហ	.548
54.36	207.5928	.574.0	2.1872	1.0565	3:9267	1.537	5.7100	3.496	12.8165
	•		•	•		,			
	0;	0	0016	.0°2	0369	0.0	0546	0.	1828
, ,	,	,			,	,			1
U		0	0101 .	0	0092	0.	0132 .	0.	0139
]				· · · · · · · · · · · · · · · · · · ·			T		

## Standard Error of Estimate

$$f(X) = C_0 + C_1 X$$
 (1)

If f(X) represents the value of Y for given values of X as estimated from (1) a measure of the scatter about the regression line of Y or X is given by the quantity

$$SY \cdot X := \frac{\sum (Y - f(X))^2}{9}$$

for small samples

$$SY.X = \frac{\Sigma (Y - f(X))^2}{N - 2}$$

is prefered by some statisticians.

N is the number of points in the data set.

If two lines, parallel to the regression line of Y or X, were constructed at vertical distances SY.X, 2SY.X, and 3SY.X from it, about 68%, 95%, and 99.7% of the sample points would be included between the two lines.

		•	•			_												. ¥6	*	.1	.93
	20 Mhz	Y = f(X) = Y - f(X)	220 . 216. 16 X 10 ⁻⁶	210 .222 144 X 10 ⁻ 6 220 .226 36 X 10	25 .226 1 x 10	225 .228 9 X 10 °	230 . 229 1 X 10 8	230 .230 0 .	230 .231   1 X 10 2	230 231 1 X 10-	225 .233 64 X 10 ⁻	240 .233 49 X 10 .	250 x 937 169 x 10 ⁻ °	255 .241 196 X 10 ⁻	248 .250 4 X.10 2	258 .266 64 X 10 ⁻⁸	755. x 10 ⁻⁶	f(x) = .1828 + .0139 N = 15	$SY.X = \frac{755 \times 10^{-6}}{13}$	$=$ , 7.62 x $10^{-3}$	= .00762
The second secon	16 Mhz	$Y = f(X) / Y - f(X)^2$			97 .096 9 x 10 6.	•	.090  .097   49 X 10	•-	•	.100   101   001.	.100 .102 4 X 10 %.		.115 .106 81 X 10		.113  .118  :25 x 10  :2	•	374. x 10 ⁻⁶	(x) =0546 +0132x = 15	$Y.X = \frac{374 \times 10^{-6}}{13}$	$= 5.36 \times 10^{-3}$	. = .00536
⟨ FREQUENCY	j3 Mhz	$Y = f(X) = Y - f(X)^2$	_0560 0650 0650.	.0555 .0627 5184 X 10 .0640 .0652 144 X 10	0680 .0656 516 x 10	.0680 .0665 225 X 10	.0680 .0667 169 x 10	.0690 .0678 144 X 10	.0720 .0689 961 X 10	.0680 .0691. 121 x 10	.0630 .0700 4900 x 10	.0730 .0704 676 X 10	.0820 .0730 8100 x 10 8	.0840 .0755 7225 X 10	.0790 .0811 441 x 10 9	.0870 .0921 2601 X 10-°	. 323.67 x 10 ⁻⁶	f(x) = .0369 + .0092X $f(x) = 15$	$SY.X = \frac{323.67 \times 10^{-6}}{13} S$	= 4.990 X 10 ⁻³	= .00499
	z Whz	$X = f(X) = Y - f(X)^2$	55 .0258, 9 X 10	.0255 .0299 16 X 10 8 .0310 .0327 289 X 10	35 .0331 16 x 10	.0350 .0341 81 X 10 8	- 64 X 10	49 X 10	.0385 .0367 324 X 10 %	.0340 .0370 900 X 10 %	.0360 .0380 400 X 10"	:0384 16 X.10	.0500 .0412 7744 X 10 %	.0525 .0440 7225 X 10 %	.0480 .0501 441 X 10 %	.0580 .0622 1764 X 107 °	193.38 X 10 ⁻⁶	f(X) = .0016 + .0101X N = 15	$SY.X = \frac{1.9338 \times 10^{-4}}{13}$	$= 3.86 \times 10^{-3}$	00386
	. ×		2.40	2.80	3.12	3.22	3.24	3.36	-	3.50	.3.60	3.64	3.92	4.20	4.80	6.00		,			

Q.

#### APPENDIX III

## ANALYTICAL TESTS FOR CLAY CONTENT

## Methylene Blue Test for Western Bentonite

Accuracy of the methylene blue test for active western bentonite, developed by Baroid Div., National Lead Co., Houston, is equal to that of the nickel sulfate method. There is little difference in the time required to make either determination; however, the equipment required for the methylene blue test is much less expensive than that required for the nickel sulfate method.

Checks showed that such common additives as dextrine, corn flour, or sea coal did not interfere with the accuracy of the test. Baroid Div. says, however, that solubilized lignite will absorb the dye and lead to high readings. The effect of lignite can be removed by boiling the sample with 3% hydrogen peroxide.

Only two brands of western and one brand of southern bentonite were tested. As in the nickel sulfate test, each brand of clay requires a separate calibration curve. Our results on southern bentonite gave a less consistent reproducibility than we obtained with the two western bentonites. Baroid Div. reports, however, that

they have tested southern bentonite without apparent difficulties.

Weigh out accurately 5 gm. sand mix into an .

Erlenmeyer flask. Add 50 ml. distilled water and boil for 5 minutes. Note that the flask will bump during boiling and will need to be held by some means. As an alternate, the flask can be shaken for 15 minutes.

Add 12 to 2 cc. of .5N sulfuric acid solution and mix thoroughly., Add methylene blue solution (1 ml = .01 milliequivalents = 3.74 gm. per liter) in increments. After each addition, shake it briefly, and, using a glass rod, place a drop of the liquid on a filter paper. If the end point has not been reached, only a blue circle will appear. If the end point has been reached, a blue-green halo will surround the blue circle.

At the first appearance of the halo, shake the solution for 2 minutes to be sure that all the methylene blue has been adsorbed. Place another drop on the filter paper. If the halo persists, the end point has been reached or passed.

A calibration curve must be made for each brand of clay By adding the equivalent amount of clay (no sand is needed) to 50 cc. of water. For example: for 1% clay in 5 gm. of sand mix, add .05 gm. of clay, and for 5% clay in 5 gm. of sand mix, add .25 gpm. of clay.

No short cuts to the test method are apparent.

Additional information on the use of this test is contained in "Determination of Cation Exchange Capacity by Methylene Blue Adsorbtion," presented by M.J. Nevins and D.J. Weintritt at a meeting of the Materials and Equipment and White Wares Division, American Ceramic Society, Bedford Springs, Pa., September 29 - October 2, 1965.

## Nickel Sulfate Test for Active Clay

The nickel sulfate method, developed in 1959 by the British Steel Casting Research Association, depends on the adsorption by active clay of nickel ions in a solution of nickel sulfate. The remaining nickel is formed into a colored complex by adding dimethyl glyoxime. Relative color intensity of the complex depends on the nickel available. Thus, the amount of active clay can be determined by measuring the intensity of the solution on a spectrophotometer at 540 m as in a standard nickel determination.

The following standard solutions are prepared:

- 1. Nickel sulfate solution-- .476 gm. NiSO, per liter of water.
- 2. Ammonium citrate solution -- 50 gm. citric acid to 100 ml. 50% ammonia.
- 3. Iodine solution (.1 Normal) -- 12.69 gm. I plus 20-25 grams KI to a liter of water.

4. Dimethyl glyoxime solution -- .1 gm. dimethyl glyoxime in 50 ml. NH,OH plus 50 ml. water.

To establish a standard curve, samples of clay weighing from .1 to .5 gm. are added to 50 ml. of nickel sulfate solution and stirred for 2 minutes. Each sample is centrifuged until a clear solution is obtained. (Note: If solutions are not clear after 30 minutes of centrifuging, they must be filtered.)

Ten ml. of the clear solutions are pipetted off and mixed with 10 ml. of ammonium citrate solution and 5 ml. of iodine solution. After this solution has stood for 3 minutes, 20 ml. of dimethyl glyoxime solution is added, and the nickel color is allowed to develop for 5 minutes.

The solution then is diluted to 200 ml. and the color measured on the spectrophotometer, using a water setting. A standard is prepared without the clay addition and tested also. The spectrophotometer readings are plotted against the weights of the clay samples as shown.

To determine the amount of active clay in a sandclay mix, samples are weighed out. For a mix in which the active clay content is anticipated to be about 2%, the sample should be about 10 gm. If as much as 10% active clay is anticipated, the sample need be only 4 gm. The is samples must be dried at 240 F before testing by the

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procedure outlined above. The spectrophotometer readings are used to determine the amount of active clay in each sample and then the percentage of active clay can be calculated.

Different brands of clay will have slightly different_calibration curves; therefore, a separate curve should be established for each brand of clay used.

#### APPENDIX IV

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