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A Comparative Analysis of Five  
Michigan Cements.

A Thesis Submitted to  
The Faculty of  
Michigan Agricultural College

By

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THESIS

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## INTRODUCTION.

The object of this Thesis is to determine the relative adaptability of five Michigan brands of Portland cement, for engineering construction; all being subjected to the same chemical and physical tests, under the same conditions, as far as possible.

The commercial names of the cements are Aetna, Burt, Michigan, Newaygo, and Peninsular. These are of particular interest to us, due to the fact that they are being used at the present time in the construction of several buildings on the M. A. C. Campus. Samples of the cements were obtained from regular trade depots in Lansing, Michigan, and the utmost care was exercised in selecting representative samples of good fresh cement in each case.

The tests were made in accordance with the Standard Specifications and Uniform Methods of Testing and Analysis for Portland cement, which have been adopted by the committees on Standard Specifications for cement of the American Society for Testing Materials. The routine of tests were as follows:

### I Specific Gravity

- (a) Le Chatelier's Specific Gravity Apparatus.

### II Fineness

- (a) Standard Sieves

### III Tensile Strength

- (a) Neat Cement

- 1- Rheile Testing Machine

IV.      Constancy of Volume

- (a) Pats in air
- (b)    "    "    water
- (c)    "    "    steam bath.

V.       Normal Consistency

- (a) Vicat Needle Apparatus
- (b) Pat test.

VI.      Time of setting

- (a) Initial
- (b) Final.

VII.     Chemical Analysis

- (a) Insoluble Residue
- (b) Chemical Constituents
- (c) Loss on Ignition.

### Specific Gravity Test.

The determination of the Specific Gravity was made with a standard LeChatelier apparatus, which is simply a flask with a graduated neck on it.

The method of procedure in each case was as follows:

The flask was filled with Benzine up to the lower mark on the neck of the flask and 64 grammes of dry cement was added slowly through a funnel. After the cement was introduced the level of the liquid rose to some division on the graduated neck.

This reading, plus 20 cu. cm. is the volume displaced by the 64 gms. of cement and the Specific Gravity equals weight of cement in gms. divided by displaced volume in cu. cm.

The results obtained were as follows:

| Specimen   | Specific Gravity |
|------------|------------------|
| Aetna-     | 3.23             |
| Burt       | 3.23             |
| Michigan   | 3.15             |
| Newaygo    | 3.12             |
| Peninsular | 3.30             |

The Specific Gravity should not fall below 3.10. In all cases it was above this value.

### Fineness.

The fineness was determined by placing fifty grammes of dried cement on a 100 sieve, which has a No. 200 sieve underneath, to these are attached a pan and cover and then moved backward and forward about 200 times per minute, at the same time striking the side gently with the palm of the hand. The operation is continued until very little cement passes through the screens. The residue on each of the screens is then weighed.

It shall leave by weight a residue of not more than 8% on the #100 sieve, and not more than 25% on the #200 sieve.

The average results obtained were as follows:

| Cement     | Residue on<br>#100 sieve | Residue on<br>#200 sieve | Amount Passing<br>#200 sieve |
|------------|--------------------------|--------------------------|------------------------------|
| Aetna      | 3%                       | 19.1%                    | 77.9 %                       |
| Peninsular | 8%                       | 25%                      | 67%                          |
| Michigan   | 2.2%                     | 24.8%                    | 73%                          |
| Burt       | 5.6%                     | 23.6%                    | 70.8%                        |
| Newaygo    | 7.9%                     | 22.02%                   | 70.08%                       |

From this data we find that the Aetna is the finest and Peninsular the coarsest cement.

### Normal Consistency.

The normal consistency was made by means of the Vicat Apparatus.

Five hundred gms. of the cement was made into a paste by kneading with the hands. It was made into a ball and placed into the larger end of a rubber ring. The cement was then placed in the Vicat Apparatus, and the needle placed at the top of the ring, and then released and allowed to settle into the cement.

Trial pastes were made with varying percentages of water, until a paste was obtained that allowed the needle to sink only 10 cm. in 30 sec.

The percentages of water used for the different cements were as follows:

| Specimen   | Water used |
|------------|------------|
| Aetna      | 23%        |
| Burt       | 21%        |
| Michigan   | 23%        |
| Newaygo    | 20%        |
| Peninsular | 19%        |

### Constancy of Volume.

The tests for Constancy of Volume comprises "normal tests," which are made in air and water, and "accelerated tests" which are made in steam.

Pats about 3 in. in dia. and  $\frac{1}{2}$  in. thick at center and tapering to a thin edge were made, and placed on clean glass plates, and stored under moist cloths for 24 hrs.

After this 24 hrs. several pats were placed in water and observed at intervals of five days each for 25 days, and all of the pats seem to lose a small amount of their hardness but not enough to cause them to distort or disintegrate.

Several other pats were placed in the air (which was as near 70 Fahr. as possible) and examined at intervals of five days each for 25 days.

All pats became very hard and firm, and the Peninsular and Michigan had the tendency of cracking in places.

A number of pats were placed in an atmosphere of steam about 1 in. above boiling water for a period of 5 hrs. and maintained at atmospheric pressure.

All specimens seemed to have a scaly appearance, but none were cracked to a very great extent.

The most significant thing found was that the Michigan and Peninsular seemed to show a tendency of cracking, disintegrating and expanding when left exposed to the atmosphere for a period of 28 days, which in our estimation,

tends to render them unsound.

The most important quality of a cement is its soundness; for no matter how high a degree of tensile strength, a cement may develop at comparatively short period, if it fails to resist the disintegrating influences of the atmosphere or water in which it is placed, it is useless as a material of construction.

This fault is usually due to an improper proportioning of the raw materials, allowing an excess of lime over what will combine with the silica and alumina of the cement mixture, or improper burning, failing to raise the temperature to a point where, all of the lime may combine with the silica and alumina, thus leaving some in an uncombined state; or from insufficient grinding of the raw materials making it impossible for all of the lime to come in contact and unite with the silica and alumina. This free or loosely combined lime on coming in contact with the moisture of the atmosphere is slacked and expands, causing the cement to crack and fall to pieces.

### Tensil Strength.

The tensil strength of cement was determined by making briquettes of neat mortar of normal consistency.

The material was weighed, placed on a non-absorbant surface and thoroughly mixed with water by kneading with the hands.

The mortar was then placed in the molds with the hands, pressed in firmly with the fingers and smoothed off with a trowel without ramming.

During the first 24 hours after molding a moist cloth was kept over the briquettes, later they were stored in a water tank.

The specimens were tested at 7, 14 and 28 days on a Rheile Briquette Testing Machine.

| Specimen | 7 days | 14 days | 28 days |
|----------|--------|---------|---------|
| Aetna    | 775    | 760     | 680     |
|          | 660    | 670     | 720     |
|          | 600    | 760     | 710     |
|          | 685    | 680     | 730     |
| Average  | 680    | 710     | 710     |
| Michigan | 490    | 620     | 780     |
|          | 600    | 620     | 620     |
|          | 670    | 550     | 690     |
|          | 620    | 700     | 620     |
| Average  | 576    | 622     | 677     |

| Specimen   | 7 days | 14 days | 28 days |
|------------|--------|---------|---------|
| Peninsular | 760    | 680     | 670     |
|            | 820    | 620     | 670     |
|            | 750    | 660     | 550     |
|            | 670    | 770     | 700     |
| Average    | 740    | 682     | 622     |
| Newaygo    | 600    | 750     | 820     |
|            | 730    | 720     | 790     |
|            | 760    | 680     | 795     |
|            | 550    | 680     | 820     |
| Average    | 660    | 707     | 806     |
| Burt       | 730    | 675     | 660     |
|            | 680    | 785     | 740     |
|            | 500    | 675     | 800     |
|            | 720    | 700     | 770     |
| Average    | 657    | 708     | 742     |

These results show that the Newaygo cement has the greatest tensile strength at 28 days and the Michigan cement the lowest tensile strength.

The minimum requirements for tensile strength for briquettes one square inch in cross section shall be 500# for 7 days and 600# for 28 days, and the cement shall show no retrogression in strength within the periods specified.

All of the cements are within the minimum requirements except the Peninsular, whose strength decreases after the seven day test.

### Time of Setting.

The time of setting was determined with a Vicat Apparatus in the following manner:

A paste of normal consistency was molded in a hard rubber ring, and placed under the needle of the Vicat Apparatus. The needle which is 1 mm. in dia. is carefully brought in contact with the paste, and then released quickly.

The cement is considered to have acquired its initial set, when the needle ceases to pass a point 5 mm. above the glass plate; and final set, when the needle does not sink visibly into the paste.

| Specimen   | Initial Set   | Final Set     |
|------------|---------------|---------------|
| Aetna      | 3 hrs. 00 min | 5 hrs. 40 min |
| Newaygo    | 2 " 40 "      | 5 " 30 "      |
| Burt       | 3 " 10 "      | 5 " 30 "      |
| Peninsular | 2 " 20 "      | 6 " 20 "      |
| Michigan   | 2 " 55 "      | 5 " 50 "      |

These results show that the Peninsular is the slowest setting cement, and the Newaygo the fastest setting cement.

The minimum requirements for time of setting is that it shall not develop initial set in less than thirty minutes, and must develop hard set in not less than one hour, nor more than ten hours.



## Chemical Analysis.

### Silica ( $\text{Si. O}_2$ )

One-half gram of cement was placed in a casserole moistened with enough water to prevent lumping, and 5 to 10 c.c. of strong HCL added, and digested with aid of gentle heat and agitation until solution was complete. The solution was then evaporated to dryness on the sand bath.

The residue is then treated to 5 to 10 c.c. HCL which is then diluted to half strength. The dish was then covered and digestion allowed to go on for 10 minutes on the bath, after which the solution was filtered and the separated silica washed thoroughly with water. The filtrate was again evaporated to dryness, the residue without further heating taken up with acid and water, and the small amount of silica it contains separated into another filter paper. The papers containing the residue were transferred wet to a weighed platinum crucible, dried, ignited, first over a Bunsen burner until the carbon of the filter paper was completely consumed, and finally over the blast for 15 minutes, and checked by further blasting or to constant weight. The following results were obtained:

| Specimen   | Percent of Silica |
|------------|-------------------|
| Peninsular | 24.20%            |
| Aetna      | 22.42%            |
| Newaygo    | 23.00%            |
| Michigan   | 23.61%            |
| Burt       | 23.03%            |

The Silica combined with lime to form calcium silica te



furnishes the active factor in the cement's hardening.

From the results obtained it can be seen that the Michigan has the greater amount of silica and the Aetna the least.

### Alumina and Iron ( $\text{Al}_2\text{O}_3$ and $\text{Fe}_2\text{O}_3$ )

The filtrate about 250 c.c. from the second evaporation for Si O was made alkaline with N H O H after adding HCL to insure 10 to 15 c.c. strong acid, and then boiled to expel excess of N H

The precipitate iron and aluminum hydrates, after settling, are washed once by decantation, and slightly on the filter. Setting aside the filtrate, the precipitate is dissolved in hot dilute HCL, the solution passing into the beaker in which the precipitation was made.

The aluminum and iron are then reprecipitated by N H O H boiled, and the second precipitate collected and washed on the same filter paper used in the first instance.

The filter paper, with the precipitate, is then placed in a weighed platinum crucible, the paper burned off and the precipitate ignited and blasted for 5 minutes, and weighed as Al O Fe, O

| Specimen   | Percent $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ |
|------------|---------------------------------------------------------|
| Burt       | 8.00%                                                   |
| Michigan   | 10.00%                                                  |
| Newaygo    | 9.00%                                                   |
| Aetna      | 7.00%                                                   |
| Peninsular | 9.04%                                                   |

These results show that the Michigan has the highest percent and Aetna cement the lowest percent of alumina and iron.

The alumina tends to quicken the setting and decrease the ultimate tensile strength.

The ferric oxides exerts very little influence upon the physical properties of the material. The dark gray color

of cement is due to the presence of iron compounds.

### Lime (Ca O )

To the combined filtrate of the  $Al_2O_3$  and  $Fe_2O_3$  precipitate a few drops  $NH_4OH$  was added and the solution brought to boiling. To the boiling solution 20 cc of a saturated solution of ammonium oxalate was added, and the boiling continued until the precipitated  $CaC_2O_4$  assumed a well-defined granular form. It was then allowed to stand until the precipitate settled, and then filtered and washed. The precipitate and filter are placed wet in a platinum crucible, and the paper burned off over a Bunsen burner. It was then ignited, redissolved in HCL and the solution made up to 100 c.c with water. Ammonia was added in slight excess, and the liquid boiled. The lime was then reprecipitated by ammonium oxalate, allowed to stand until settled, filtered and washed. The precipitate with filter paper was ignited and blasted in a covered crucible to constant weight.

The following results were obtained:

| Specimen   | Ca O   |
|------------|--------|
| Aetna      | 60.00% |
| Michigan   | 60.49% |
| Peninsular | 61.40% |
| Newaygo    | 61.23% |
| Burt       | 63.12% |



## Magnesia (Mg O)

The combined filtrates from the calcium precipitates were acidified with HCL and concentrated on the steam bath to about 150 c c, 10 c c of saturated solution of  $\text{Na (N H}_4\text{) H P O}_4$  was added, and the solution boiled for several minutes.

It was then removed from the flame and cooled in ice water. After cooling,  $\text{N H O H}$  is added drop by drop with constant stirring until the crystalline ammonium-magnesium ortho-phosphate began to form, the stirring being continued for several minutes.

It was then set aside for several hours in a cool atmosphere and then filtered. The precipitate is redissolved in hot dilute HCL the solution made up to 100 c.c. 1 cc of saturated solution  $\text{Na (N H}_4\text{) H P O}_4$  added, add ammonia drop by drop, with constant stirring until the precipitate is again formed as described and the ammonia in slight excess.

It was then allowed to stand for about two hours when it was filtered on a paper, ignited, cooled and weighed.

The following results were obtained:

| Specimen   | Mg. O  |
|------------|--------|
| Aetna      | 3.24 % |
| Michigan   | 2.82 % |
| Burt       | 2.81 % |
| Peninsular | 2.00 % |
| Newaygo    | 3.57 % |

The maximum amount of Magnesia allowable is 4%. All of the cements contained less than this amount.



Sulphuric Acid  
(  $\text{S O}_3$  )

One gram of the cement was dissolved in 15 c c of HCL, filtered and the residue wasted thoroughly. This solution was made up to 250 c c in a beaker and boiled. To the boiling solution 10 cc of saturated solution of  $\text{Ba Cl}_2$  was added slowly drop by drop from a pipette and the boiling continued until the precipitate was well formed. It was then set aside over night, or for a few hours, filtered, ignited, and weighed as  $\text{Ba S O}_4$ .

The following percentages of  $\text{S O}_3$  were obtained:

| Specimen   | Percent of $\text{S O}_3$ |
|------------|---------------------------|
| Aetna      | 1.65                      |
| Michigan   | 1.63                      |
| Newaygo    | 1.68                      |
| Burt       | 1.68                      |
| Peninsular | 1.75                      |

According to these results the Peninsular has highest percentage of  $\text{S O}_3$  (1.75) and Michigan lowest of (1.63)

The maximum allowed by specification is 1.75 and all fall at or below this amount.

### Ignition Test.

Half a gram of cement was weighed out in a platinum crucible, placed in a hole in an asbestos board so that about  $3/5$  of the crucible projects below, and blasted for 15 minutes. The loss by weight, which is checked by a second blasting of 5 minutes, is the loss on ignition.

In all cases the loss was less than .5 of 1%.

# Chemical Analysis

|                         | Aetna        | Michigan     | Newaygo      | Burt         | Peninsular   |
|-------------------------|--------------|--------------|--------------|--------------|--------------|
| Silica                  | 22.42        | 23.61        | 23.00        | 23.03        | 24.20        |
| Alumina & iron          | 7.00         | 10.00        | 9.02         | 8.00         | 9.04         |
| Magnesia                | 3.24         | 2.52         | 3.57         | 2.81         | 2.00         |
| Sulphur Trioxide .      | 1.65         | 1.63         | 1.68         | 1.68         | 1.75         |
| Lime                    | <u>60.00</u> | <u>60.44</u> | <u>61.23</u> | <u>63.12</u> | <u>61.40</u> |
|                         | 99.17        | 99.50        | 98.50        | 98.64        | 98.39        |
| Amount unaccounted for- | .83          | .50          | 1.50         | 1.36         | 1.61         |

### Conclusion.

In summing up the results of our analysis of the various cements, we find that several of the samples are nearly on a par.

In the Specific Gravity tests, our results ranged from 3.12 to 3.30 and all being over the required amount of 3.10, we considered that none had been adulterated.

The results of our fineness tests showed the following in order of fineness, Aetna, Michigan, Burt, Newaygo and Peninsular.

The fineness of the material is a measure of its cementing value, and a fine cement accordingly will be much stronger when mixed in a mortar, or it can be mixed with a larger portion of sand than a coarse one, and yet attain the same strength.

A test for fineness is nearly always included in cement specifications, as the indications from a fair degree of fineness coupled with proper tensile strength, neat, are that the cement will give good results, when mixed with sand.

The tensile strength test gave best results for the Newaygo, with the Burt, Aetna, Michigan and Peninsular next in order.

In reality, the neat break is not of so much value as we are apt to suppose, and taken by itself it is a little criterion of the quality of cement, Unsound cement often gives notoriously high results and the addition of plaster or gypsum will also increase the neat strength. In both these instances there is apt to be on long time breaks a falling off

of strength, permanent in the former case, and usually only temporary in the latter case.

The Peninsular cement gave very high results for the 7-day test, but showed a marked decrease in the 14 and 28 day tests. According to the Standard Specifications no cement shall show retrogression in strength within periods specified.

In the time of setting tests the results for the initial set varied from 3 hours to 2 hours, 55 minutes, and the final set from 5 hrs. 30 minutes to 6 hrs. and 20 minutes.

According to the Standard Specifications it shall not develop initial set in less than thirty minutes and must develop hard set in not less than one hour, nor more than ten hours.

The rapidity with which a cement sets furnishes us with no indication of its strength. The test is usually made to determine the fitness of a material for a given piece of work. The slow-setting cements can be mixed in larger quantities than quick-setting, and do not have to be handled so quickly.

The Aetna cement ranks first in fineness, third in tensile strength, and second in length of time of setting. It has a low percent of silica and lime which tends to decrease the strength. The percent of Magnesia is quite large which decreased the strength.

The Burt cement ranks third in fineness, second in tensile strength and first in length of time of setting. It has a largest percent of lime and silica which are the

two main strength producing constituents of cement. It has a very low percent of Magnesium which is a defective agent in producing cracking and disintegration.

The Michigan cement ranks second in fineness, fourth in strength and third in length of time of setting. It has a very small percent of lime and silica and an extra large amount of iron and alumina in it. It also tends to be unsound judging from the constancy of Volume Test.

The Newaygo cement ranks first in tensile strength fourth in fineness, fourth in length of setting. It has a large percent of lime and silica and also a large percent of magnesium the latter being a very harmful constituent.

The Peninsular cement ranks last in tensile strength last in fineness, and last in length of time of setting. It contains a large percentage of alumina and iron and sulphur. It also showed a retrogression in tensile strength after seven days, and gave very poor results in soundness test.

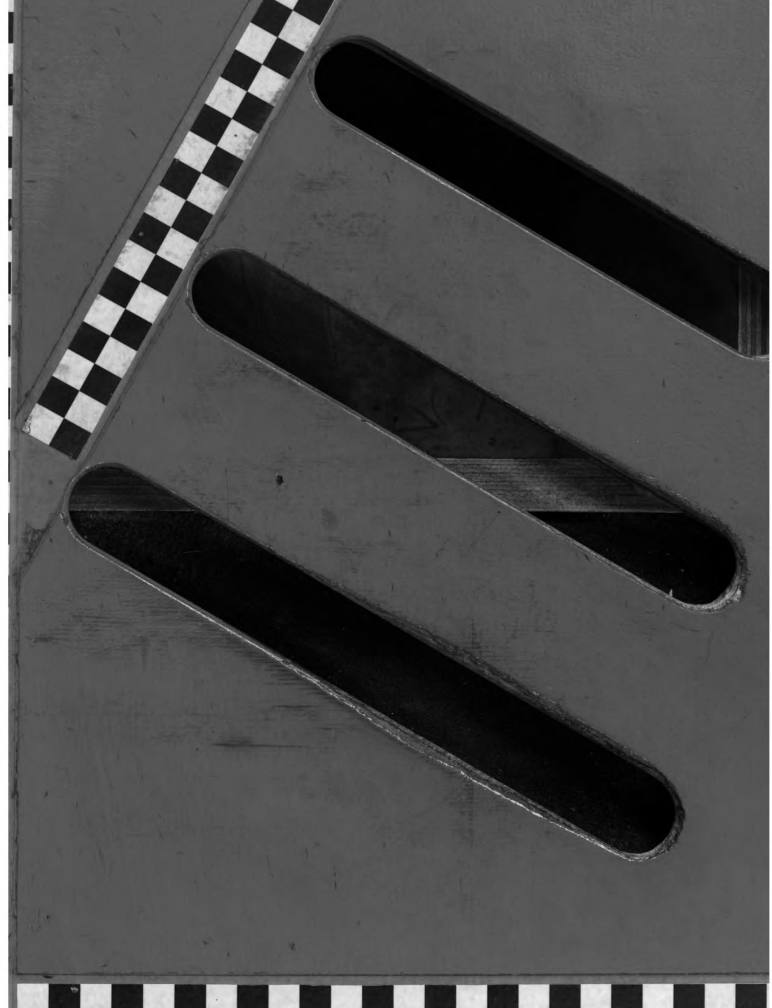
Taking into consideration the above results, we believe that the Burt Cement is the best for building construction, because it contains more of the good qualities mentioned at the beginning of this article, although the Aetna and Newaygo also have many good points in their favor and could be used with very good results.



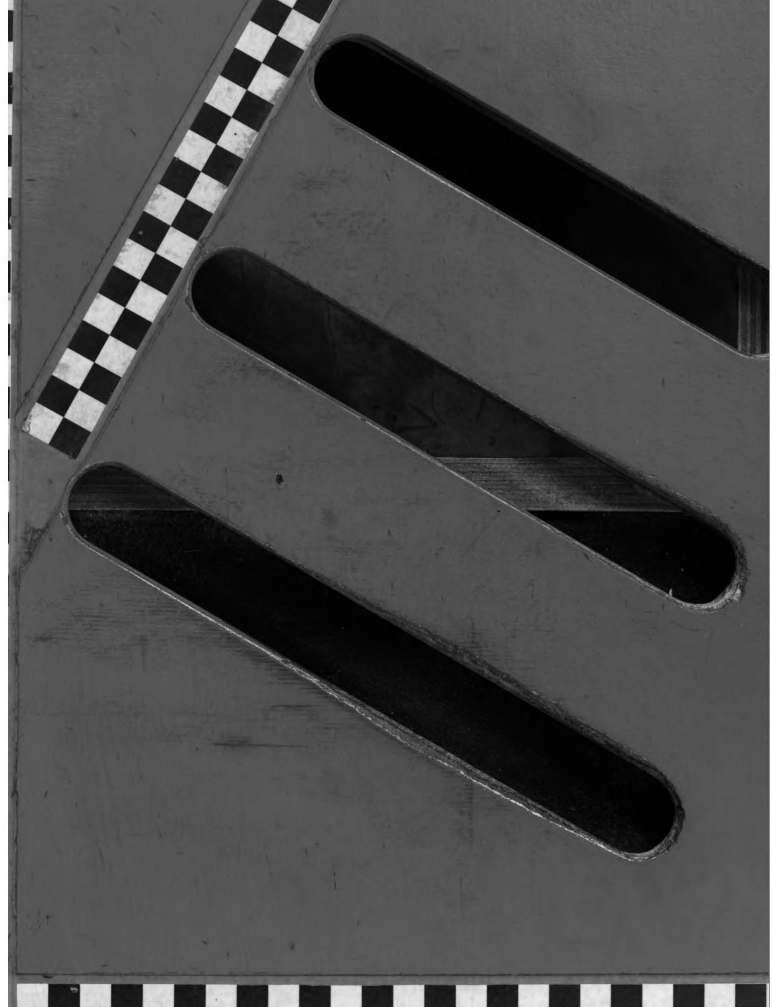
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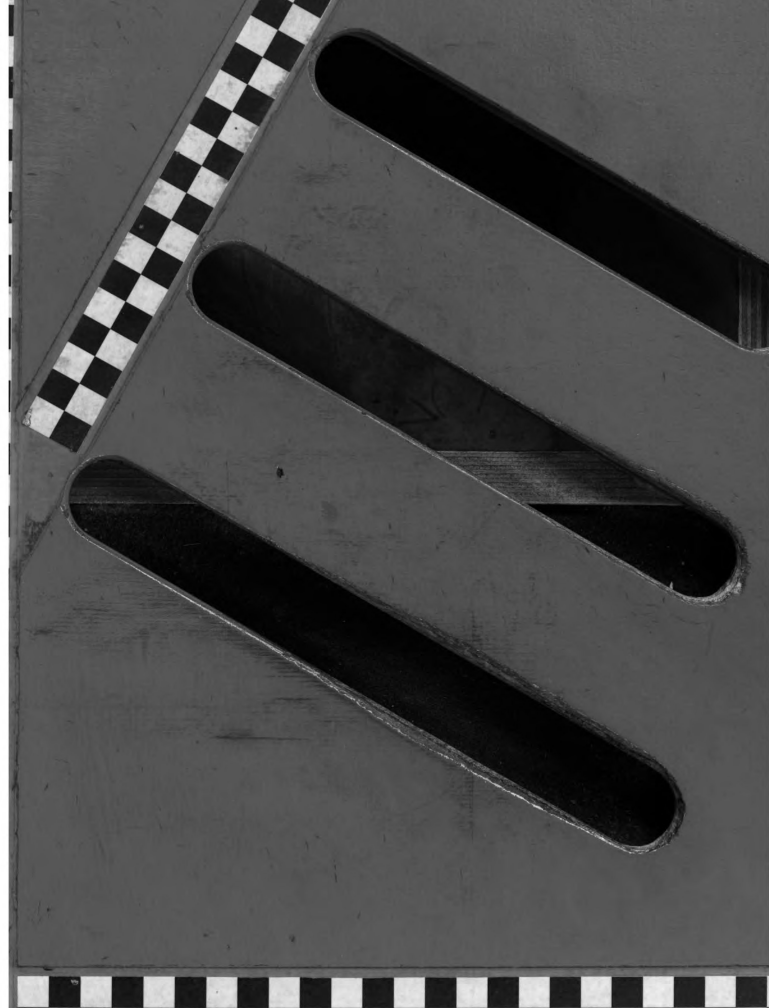












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