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SC-CR-68-3443

Jan 1968

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VIA: AIR MAIL

January 17, 1968

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Sandia Corporation
P.O. Box 5800
Albuquerque, New Mexico 87115

Attention: Mr. F. Duggin, Jr., Buyer
Mr. N. Rosenberg, Requester

Reference: Purchase Order Number 58-2496

Subject: Progress Report Number 4
December 1, 1967 to January 1, 1968

[redacted]

Gentlemen:

The following work was accomplished during the reporting period:

0 Abstract

1.1 When the Ti:$CaCrO_4$ ratio was varied in papers and slurries, the results were proportional.

1.2 The slurries had about 75 cal/gm higher heat value than 12% fiber paper.

1.3 Only a slight fiber reaction appears to take place with Ti:$CaCrO_4$.

1.4 Ti:$CaCrO_4$ heat papers were found to be unacceptable due to the low electrical resistance during and after burning.

1.5 The Ti:$CaCrO_4$ paper 16:84 had an order of magnitude lower electric resistance than standard Zr:$BaCrO_4$ 23:72 (14.8 vs 227 ohms).

1.6 Silicon was insensitive in tests to date.

1.7 Cleaning the silicon powders or using active oxidizers also produced no improvement.

1.8 The addition of Ti as a sensitizer aided the silicon reaction.

SANDIA SYSTEMATIC DECLASSIFICATION REVIEW
Determination (Circle Numbers): 1. Classification Retained 2. Classification Changed to: 3. Contains No DOE Classified Information 4. Coordinate With: 5. Contains UCNI 6. Comments: not trust instrat before to approved
1st Review Date: 12-14-95 Authority: BADC ADD Name: W. Lange
2nd Review Date: 1/5/96 Authority: ADD Name: PB Cramer

SANDIA SYSTEMATIC DECLASSIFICATION REVIEW
DOWNGRADING OR DECLASSIFICATION STAMP
CLASSIFICATION CHANGED TO: U AUTHORITY: R.B. Cramer
PERSON CHANGING MARKING & DATE: Emelia Sepa 05-21-96 RECORD ID: 95SN019
PERSON VERIFYING MARKING & DATE: W Lange 6-26-96 DATED: 01-05-96

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DOCUMENT No. 0-41141
1-18-68

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## 2.0 Summary of Accomplishments

### 2.1 $Ti:CaCrO_4$ Slurries

12% Fibers reduced the heat value by 70 cal/gm. Slight fiber reaction appears to take place as Ti was increased (10 cal/gm at 40% Ti). Figure I is a plot of the $Ti:CaCrO_4$ ratio vs. heat value as a slurry as well as 12% fiber paper. The slurry and heat paper curves are proportional. The heat value of the slurries and paper were slightly higher than calculated (See Report I stoichiometric calculations). See Chart I for details of slurry data and appendix for slurry procedure.

### 2.2 Heat Paper Resistance

$Ti:CaCrO_4$ heat papers were found to be unacceptable due to their low resistance during and after burning. When the amount of Ti was decreased, the resistance increased. A ratio of 16:84, $Ti:CaCrO_4$, had the highest resistance studied before, during and after burning. See Chart II for detailed data.

Figure II is a comparison of standard $Zr:BaCrO_4$ 28:72, vs $Ti:CaCrO_4$, 16:84 paper resistance while burning. The $Ti:CaCrO_4$ had an order of magnitude lower resistance. See Chart III.

The resistance measurements before and after burning were repeated using a 1650A Radio Corporation Impedance Bridge. This was done because the circuit as originally designed would only read between 0.5 to 20,000 ohms. Before ignition, the resistance of $Ti:CaCrO_4$ was found to be similar to standard $Zr:CaCrO_4$ below 25% Ti. See Figure III. After $Zr:BaCrO_4$ paper burns, the resistance returns to pre-ignition levels. The $Ti:CaCrO_4$ stays very conductive at high Ti concentrations and slightly conductive at low concentrations.

Figure 4 is a plot of the log of the resistance vs. various percentages of Ti. The plot is a linear function.

### 2.3 Silicon Evaluation

Silicon was found to be insensitive when used in pyrotechnic powders and papers. Cleaning the silicon powder with alcohol-acetone, $H_2SO_4$, or HF did not improve sensitivity. Suppliers informed us that a finer particle size or more sensitive silicon powders are not available. Oxidizers $MnO_2$, $KClO_4$, and $V_2O_5$ were found insensitive when silicon was used. Only the

higher silicon ratio's with $KClO_4$ burned.

Initial work was done in sheet molds. When they failed to burn, slurries and dry powders were used to speed up the investigation. Chart IV is a synopsis of the papers tested. Chart V shows powder mixes, Si cleaning method, oxidizers and blending methods tried.

Addition of Ti to the silicon powder acted as a sensitizer. See Charts IV and V-Ti:Si blends.

3.0 Future Work

3.1 Study of the effects of

1. Percent of fibers on paper resistance

2. Diluents on paper resistance

3. Varying the types of fibers on paper resistance. Vitron will be replaced by asbestos, kaowool, PKT, and other fibers.

4. On resistance of equivalent Silicon weights added to $Ti:CaCrO_4$ heat paper

5. On ignition sensitivity of Ti on $Si/CaCrO_4$ paper

3.2 A Ti oxidizer study will be initiated using $MnO_2$, $V_2O_5$, $CaSO_4$, and others.

3.3 Ignition sensitivity of $Ti:CaCrO_4$ heat paper will be completed. (Study delayed due to circuit materials not being received from vendor.)

Approved By:

CATALYST RESEARCH CORPORATION

H. J. Goldsmith

H. J. Goldsmith, Manager
Research & Development Div.

Submitted By:

CATALYST RESEARCH CORPORATION

G. C. Bowser

G. C. Bowser
Project Engineer

[redacted]



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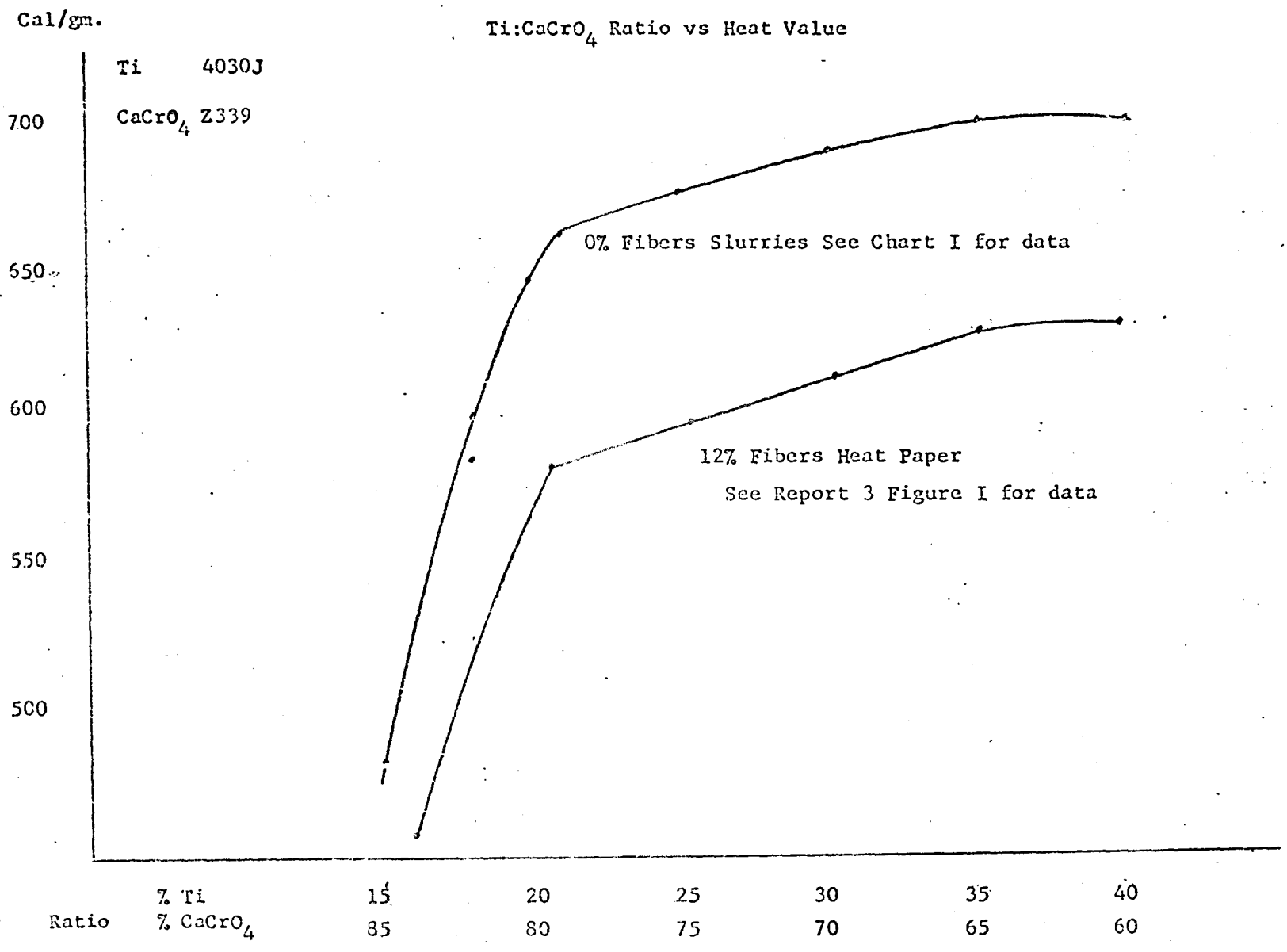
Figure I

Ti:$CaCrO_4$ Ratio vs Heat Value

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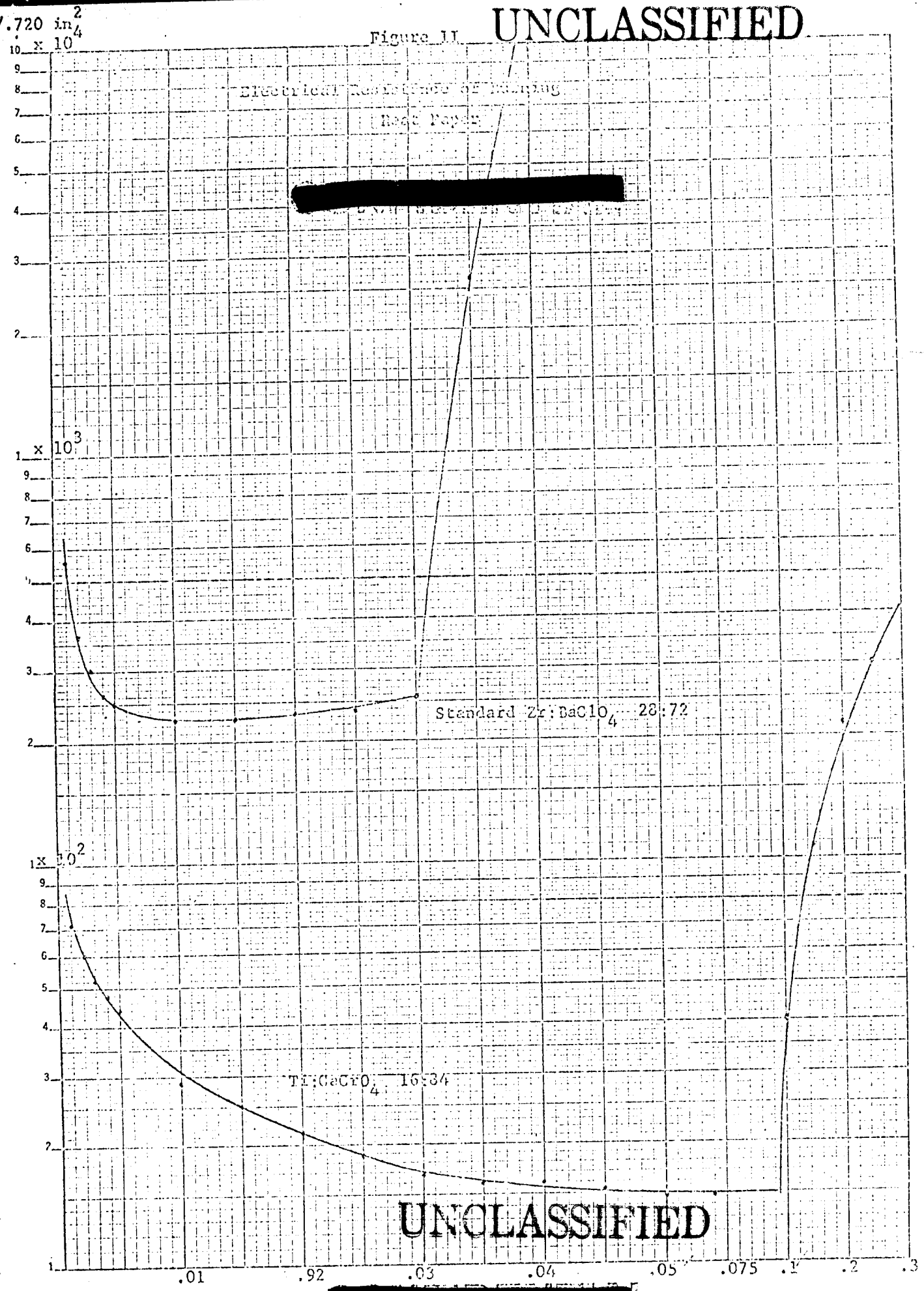


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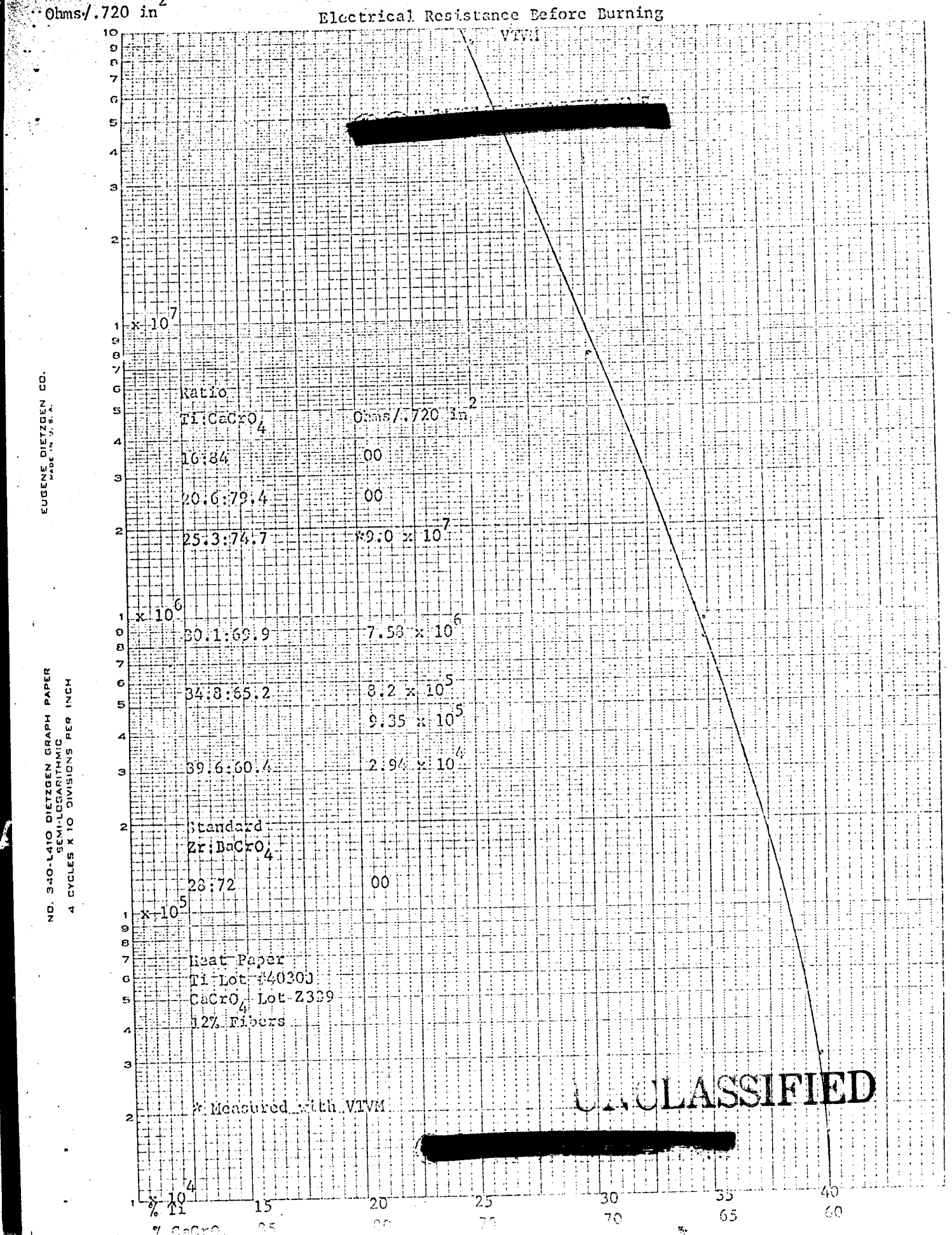
FIGURE III

Electrical Resistance Before Burning

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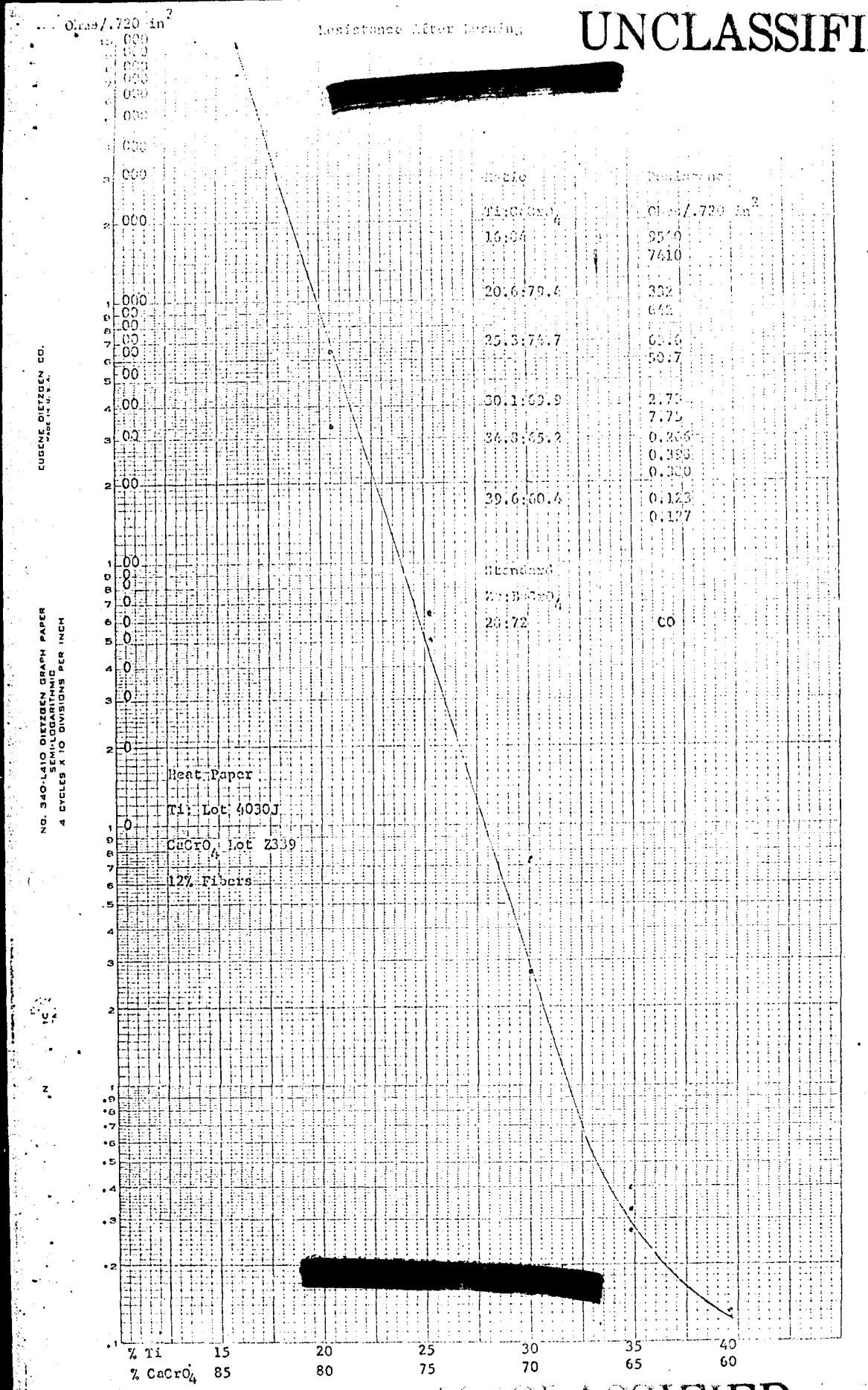


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CHART I

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Ti:$CaCrO_4$ Heat Values (Slurries)

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| Ratio Ti:$CaCrO_4$ | Free Ti | Impurities | Grams Ti | True $CaCrO_4$ | Impurities | Grams $CaCrO_4$ | CC Water | %Solids | Cup Wt. | Sample Wt. | Cal/gm. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 15:85 | 15.000 | 0.924 | 15.924 | 85.000 | 2.233 | 87.233 | 75 | 57.1 | 61.4327 | 1.9930 | 479.62 |
| | | | | | | | | | 58.7047 | 2.2244 | 487.73 |
| | | | | | | | | | 62.0428 | 2.0312 | 481.96 |
| | | | | | | | | | | | 483.10 |
| 20:80 | 20.000 | 1.231 | 21.231 | 80.000 | 2.102 | 82.102 | 70 | 58.8 | 60.5179 | 2.1875 | 639.77 |
| | | | | | | | | | 59.7427 | 2.1382 | 645.66 |
| | | | | | | | | | 61.1658 | 1.9590 | 645.82 |
| | | | | | | | | | 62.0772 | 2.0024 | 652.58 |
| | | | | | | | | | 60.5099 | 1.9724 | 644.61 |
| | | | | | | | | | | | 645.69 |
| 25:75 | 25.000 | 1.539 | 26.539 | 75.000 | 1.970 | 76.970 | 70 | 58.8 | 60.1085 | 2.1141 | 665.18 |
| | | | | | | | | | 61.9690 | 2.0991 | 676.14 |
| | | | | | | | | | 61.2070 | 2.0802 | 685.91 |
| | | | | | | | | | 59.7430 | 2.0045 | 666.87 |
| | | | | | | | | | 58.7050 | 1.9714 | 681.60 |
| | | | | | | | | | | | 675.14 |
| 30:70 | 30.000 | 1.847 | 31.847 | 70.000 | 1.839 | 71.839 | 70 | 58.8 | 60.2682 | 2.0970 | 656.71 |
| | | | | | | | | | 60.4758 | 2.0423 | 692.14 |
| | | | | | | | | | 60.5530 | 2.0152 | 686.09 |
| | | | | | | | | | | | 689.12 |
| 35:65 | 35.000 | 2.155 | 37.155 | 65.000 | 1.708 | 66.708 | 70 | 58.8 | 61.1550 | 2.0715 | 693.99 |
| | | | | | | | | | 60.1065 | 2.0272 | 702.83 |
| | | | | | | | | | 61.2070 | 2.0055 | 699.13 |
| | | | | | | | | | | | 698.65 |
| 40:60 | 40.000 | 2.463 | 42.463 | 60.000 | 1.576 | 61.576 | 70 | 58.8 | 59.7410 | 2.0357 | 700.52 |
| | | | | | | | | | 60.5115 | 2.0590 | 699.22 |
| | | | | | | | | | 61.9710 | 2.0337 | 698.40 |
| | | | | | | | | | | | 699.38 |

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CHART I (Continued ---)

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$Ti:CaCrO_4$ Heat Values (Slurries)

| Ratio $Ti:CaCrO_4$ | Free Ti | Impurities | Grams Ti | True $CaCrO_4$ | Impurities | Grams $CaCrO_4$ | CC Water | %Solids | Cup Wt. | Sample Wt. | Cal/gm. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 18:82 | 18.000 | 1.108 | 19.108 | 82.000 | 2.154 | 84.154 | 70 | 58.8 | 60.2655 | 2.0161 | 584.10 |
| | | | | | | | | | 60.4693 | | |
| | | | | | | | | | 61.5245 | 2.0277 | 599.24 |
| 21:79 | 21.000 | 1.293 | 22.293 | 79.000 | 2.076 | 81.076 | 70 | 58.8 | 62.0391 | 2.0277 | 656.04 |
| | | | | | | | | | 61.1540 | 1.8412 | 661.64 |
| | | | | | | | | | 58.7015 | 2.0335 | 666.97 |
| | | | | | | | | | | | 661.55 |

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CHART II

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Resistance of Heat Paper (Sanborn Recorder)

| Fuel:Oxidizer | Before | | | During | | | After | | |
|---|---|---|---|---|---|---|---|---|---|
| Ratio | Amp | Voltage | Ohms | Amp | Voltage | Ohms | Amp | Voltage | Ohm |
| Standard | > 20,000 | | | .044 | 10.2 | 231.8 | > 20,000 | | |
| $Zr:BaCrO_4$ | | | | | | | | | |
| 28:72 | | | | | | | | | |
| $Ti:CaCrO_4$ 39.6:60.4 | .048 | 11.2 | 233.3 | < 0.5 | | | <0.5 | | |
| | .025 | 15.3 | 612.0 | " | | | " | | |
| 34.8:65.2 | .015 | 16.3 | 108.7 | " | | | " | | |
| 30.1:69.9 | > 20,000 | | | " | | | " | | |
| 25.3:74.7 | " | | | .216 | 0.3 | 1.4 | .160 | 4.2 | 26.3 |
| 20.6:79.4 | " | | | .200 | 0.7 | 3.50 | .072 | 12.3 | 170.8 |
| | " | | | .196 | 0.7 | 3.56 | .040 | 15.2 | 381.0 |
| 16:84 | " | | | .184 | 2.0 | 10.9 | .006 | 19.4 | 3233 |
| | " | | | .176 | 2.6 | 14.2 | 0039 | 19.6 | 5025 |

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CHART III

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Resistance of Burning Heat Paper

Standard Zr:$BaCrO_4$ 28:72

| Time Seconds | Amp. | Voltage | Ohms. |
|---|---|---|---|
| .01 | .025 | 14.0 | 560.0 |
| .02 | .035 | 12.8 | 365.7 |
| .03 | .040 | 12.0 | 300.0 |
| .04 | .041 | 11.3 | 261.5 |
| .05 | .043 | 10.7 | 250.0 |
| .10 | .044 | 10.0 | 227.0 |
| .15 | .044 | 10.0 | 227.0 |
| .20 | .044 | 10.2 | 232.8 |
| .25 | .044 | 10.5 | 238.6 |
| .30 | .043 | 11.0 | 255.8 |
| .35 | .007 | 18.6 | 2,657.1 |
| .40 | .002 | 19.5 | 9,750.0 |
| .45 | .001 | 19.8 | 19,800 |
| .50 | .0005 | 19.9 | 39,800 |

Ti:$CaCrO_4$ 16:84

| | | | |
|---|---|---|---|
| .01 | .112 | 8.0 | 71.3 |
| .02 | .120 | 7.2 | 60.0 |
| .03 | .125 | 6.6 | 52.8 |
| .04 | .130 | 6.2 | 47.6 |
| .05 | .136 | 6.0 | 44.1 |
| .10 | .152 | 4.4 | 28.9 |
| .15 | .160 | 4.0 | 25.0 |
| .20 | .168 | 3.6 | 21.4 |
| .25 | .170 | 3.2 | 18.8 |
| .30 | .172 | 2.9 | 16.8 |

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CHART III (Continued ---)

Ti:$CaCrO_4$ 16:84

| Time Seconds | Amp. | Voltage | Ohms. |
|---|---|---|---|
| .35 | .174 | 2.8 | 16.0 |
| .40 | .174 | 2.8 | 16.0 |
| .45 | .176 | 2.7 | 15.3 |
| .50 | .176 | 2.6 | 14.8 |
| .70 | .176 | 2.6 | 14.8 |
| .100 | .136 | 5.5 | 40.4 |
| .150 | .094 | 10.2 | 108 |
| .200 | .060 | 12.8 | 213.4 |
| .250 | .048 | 14.4 | 300.0 |



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CHART IV HEAT PAPER

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| Silicon | | | | | Oxidizers | | | |
|---|---|---|---|---|---|---|---|---|
| | Assay | Fisher | Micro-Merograph | Gain on Ignition | Oxidizer | Lot No. | Assay | Particle Size |
| Hummel 4 μ cleaned with Acetone and Alcohol | 98.5% | 4.0 | SA-6 | | $CaCrO_4$ | Z339 | 97.44 | 2.356 |
| Hummel 4 μ cleaned with $H_2SO_4$ | 98.5% | 4.0 | SA-6 | | $CaCrO_4$ | Z339 | 97.44 | 2.356 |
| Hummel 4 μ cleaned with HF | 98.5% | 4.0 | SA-6 | | $CaCrO_4$ | Z339 | 97.44 | 2.356 |
| Hummel 4 μ (Not Cleaned) | 98.5% | 4.0 | SA-6 | | $CaCrO_4$ | Z339 | 97.44 | 2.356 |
| Hummel 9 μ cleaned with Acetone and Alcohol | 98.5% | 9.0 | SA-5 | | $CaCrO_4$ | Z339 | 97.44 | 2.356 |
| Fairmont 45 μ cleaned with Acetone & Alcohol | 98.64% | 5.0 | SA-7 | | $CaCrO_4$ | Z339 | 97.44 | 2.356 |
| Crane (Not Cleaned) | 98% | | | | $CaCrO_4$ | Z339 | 97.44 | |
| Hummel 4 μ (not cleaned) | 98.5% | 4.0 | SA-6 | | $MnO_2$ | 31068 | 99.7 | |
| Hummel 4 μ (not cleaned) | 98.5% | 4.0 | SA-6 | | $V_2O_5$ | - | 99.9 | - |
| Hummel 4 μ (not cleaned) | 98.5% | 4.0 | SA-6 | | $KLLO_4$ | - | - | - |
| Hummel 4 μ (not cleaned | 98.5% | 4.0 | SA-6 | | $CaCrO_4$ | Z339 | 97.44 | 2.356 |
| Titanium | 94.2% | 2.2 | | | | | | |

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CHART IV HEAT PAPER (Continued)

HEAT PAPER

| | Si - Oxidizer Ratio | 6% Vitron 6% Fiber Frax | Free Si | Impurities | Free Oxidizer | Impurities | Solvility Adjustment | Cal/GM | Burning Rate | Firing Tube | Price |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Hummel 4μ cleaned with Acetone and Alcohol | 21.2/78.8 | 12% | 18.66 | .28 | 69.34 | 1.82 | 8.05 | | | | $7.50 |
| Hummel 4μ cleaned with $H_2SO_4$ | 21.2/78.8 | 12% | 18.66 | .28 | 69.34 | 1.82 | 8.09 | | | | $7.50 |
| Hummel 4μ cleaned with HF | 21.2/78.8 | 12% | 18.66 | .28 | 69.34 | 1.82 | 8.05 | | | | $7.50 |
| Hummel 4μ (Not Cleaned) | 21.2/78.8 | 12% | 18.66 | .28 | 69.34 | 1.82 | 8.05 | | | | $7.50 |
| Hummel 9μ cleaned with Acetone and Alcohol | 21.2/78.8 | 12% | 18.66 | .28 | 69.34 | 1.82 | 8.05 | | | | $7.50 |
| Fairmont 45μ cleaned with Acetone & Alcohol | 21.2/78.8 | 12% | 18.66 | .26 | 69.34 | 1.82 | 8.05 | | | | FREE |
| Crane (Not Cleaned) | 21.2/78.8 | 12% | 18.66 | .38 | 69.34 | 1.82 | 8.05 | | | | FREE |
| Hummel 4μ(Not cleaned) | 23.4/76.6 | 12% | 20.59 | .31 | 67.41 | 0.20 | - | | | | |
| Hummel 4μ(Not cleaned) | 30.71/57.23 | 12% | 30.71 | .47 | 57.23 | 0.06 | - | | | | |
| Hummel 4μ(Not cleaned) | 38.02/49.95 | 12% | 38.02 | .58 | - | - | * | | | | |
| Hummel 4μ(Not cleaned) | | 12% | 8.13 | .12 | 60.28 | 1.58 | 8.05 | 602.87 | 2.01 | Burned | |
| Titanium | | | 13.82 | .85 | | | | | | | |

*No solubility correction was made because saturated solution of $KClO_4$ was used

(8.13 Si + 13.82 Ti) : 60.28 $CaCrO_4$

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CHART V

SILICON OXIDIZER BLENDS

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| Supplier | Wt. % Si | Si Cleaning Method | Oxidizer | Wt. % | (Blending Method) | Results |
|---|---|---|---|---|---|---|
| Hummel 4 μ | 43.4% | None | $KClO_4$ | 56.8% | Slurry | Did not Burn |
| Hummel 4 μ | 23.4% | None | $MnO_4$ | 76.6% | Slurry | Did not Burn |
| Hummel 4 μ | 34.9% | None | $V_2O_5$ | 65.1% | Slurry | Did not Burn |
| Hummel 4 μ | 20% | None | $KClO_4$ | 80% | Dry Mix A | Did not Burn |
| Hummel 4 μ | 30% | None | $KClO_4$ | 70% | Dry Mix A | Did not Burn |
| Hummel 4 μ | 75% | None | $KClO_4$ | 25% | Dry Mix B | Did Burn |
| Hummel 4 μ | 85% | None | $KClO_4$ | 15% | Dry Mix B | Did Burn When Ignited With Heat Paper |
| Hummel 4 μ | 5% | $H_2SO_4$ | $KClO_4$ | 95% | Dry Mix B | Did not Burn |
| Hummel 4 μ | 20% | $H_2SO_4$ | $KClO_4$ | 80% | Dry Mix B | Did not Burn |
| Hummel 4 μ | 30% | $H_2SO_4$ | $KClO_4$ | 70% | Dry Mix B | Did not Burn |
| Hummel 4 μ | 50% | $H_2SO_4$ | $KClO_4$ | 50% | Dry Mix B | Did not Burn |
| Hummel 4 μ | 75% | $H_2SO_4$ | $KClO_4$ | 25% | Dry Mix B | Did Burn |
| Hummel 4 μ | 30% | HF | $KClO_4$ | 70% | Dry Mix B | Did not Burn |
| Hummel 4 μ | 50% | HF | $KClO_4$ | 50% | Dry Mix B | Did not Burn |
| Hummel 4 μ | 75% | HF | $KClO_4$ | 25% | Dry Mix B | Did Burn When Ignited With Heat Paper |

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CHART V (Continued ---)

SILICON OXIDIZER BLENDS

| Supplier | Wt. % Si | Si Cleaning Method | Oxidizer | Wt. % | (Blending Method) | Results |
|---|---|---|---|---|---|---|
| Crane | 50% | None | $KCLO_4$ | 50% | Dry Mix B | Did not Burn |
| Crane | 75% | None | $KCLO_4$ | 25% | Dry Mix B | Did Burn When Ignited With Heat Paper |
| Hummel 4 μ | 15% | None | $MnO_2$ | 85% | Dry Mix A | Did not Burn |
| Hummel 4 μ | 20% | None | $MnO_2$ | 80% | Dry Mix A | Did not Burn |
| Hummel 4 μ | 25% | None | $V_2O_5$ | 75% | Dry Mix B | Did not Burn |
| Hummel 4 μ | 34.9% | None | $V_2O_5$ | 65.1% | Dry Mix B | Did not Burn |
| Hummel 4 μ | 75% | None | $V_2O_5$ | 25% | Dry Mix B | Did not Burn |
| Hummel 4 μ | 20.88% | None | $Fe_2O_3$ | 79.12% | Dry Mix B | Did not Burn |
| Hummel 4 μ | 50% | None | $Fe_2O_3$ | 50% | Dry Mix B | Did not Burn |
| Hummel 4 μ | 10% | None | $Fe_2O_3$ | 90% | Dry Mix B | Did not Burn |
| Si Hummel 4 μ<br>Ti 4030J | 8.79%<br>16.48% | None | $CaCrO_4$ | 74.73% | Dry Mix A | Did Burn (Well) |

*Slurries were made by mixing the prescribed weight ratios of Si to oxidizer + several Ml. of water in a morter and pestle.

Dry Mixes method A were made by mixing the prescribed weight ratios of Si to oxidizer with a steel rule.

Dry mixes method B were made by mixing the prescribed weight ratios of Si to oxidizer in a morter and pestle.

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APPENDIX

Slurry Procedure

Weigh out 100 grams of the desired powder to the desired ratio (corrected for impurities) to the nearest mg. Place powder in blender with 70 to 75 ml. of water. Blend for 2 minutes. Calculate percent solids. Take sample with glass tube while mixing. Place pre-weighed Parr cup on balance and add sample (2 grams of solids). Make sure no slurry is on side of cup. Dry sample over night at 150°F in a forced air oven. Cool, reweigh, and calculate sample weight. Check heat value.

Resistance Measuring Procedure

Apparatus Design

A Device was developed to measure the electrical resistance of heat paper before, during, and after burning. The instrument consists of two iron electrodes supported by an insulated fixture. The fixture is placed in a Carver Press. Figure 6 shows the assembled apparatus ready for testing.

Circuit Design

A regulated power supply is connected to the electrodes and set at 20.0 Volts. Channel "D" of the recorder is placed across the electrodes to measure the heat paper. A 100 ohm resistor is placed in the circuit and the voltage drop across the resistor is read on channels "A" and "B" of the recorder. Channels "A" and "B" are calibrated to read high - low current levels of the circuit. Figure 7 is the wiring diagram used.

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## Procedure

A heat pad 1-3/16" diameter is placed between electrodes with a surface area of .720 $in^2$. The paper is pressed at 500 lbs. in a Carver Press. The recorder is turned on and the paper is ignited. The resistance of the paper is calculated from the voltage drop on Channel "D" and the current from Channels "A" and "B". A resistance range of 0.5 to 20,000 ohms can be read.

## Measurements Before and After Burning

The above circuit was modified by placing a Radio Corporation 1650A Impedence Bridge to the electrodes. The paper was pressed at 500 lbs. and the resistance before ignition was measured. The paper was ignited, then allowed to cool for 5 minutes. The Carver Press was reset at 500 lbs. and the resistance read again. The heat paper resistance is corrected for the resistance of the electrodes and wire. A range of $2 \times 10^{-3}$ to $11 \times 10^{6}$ ohms can be read.

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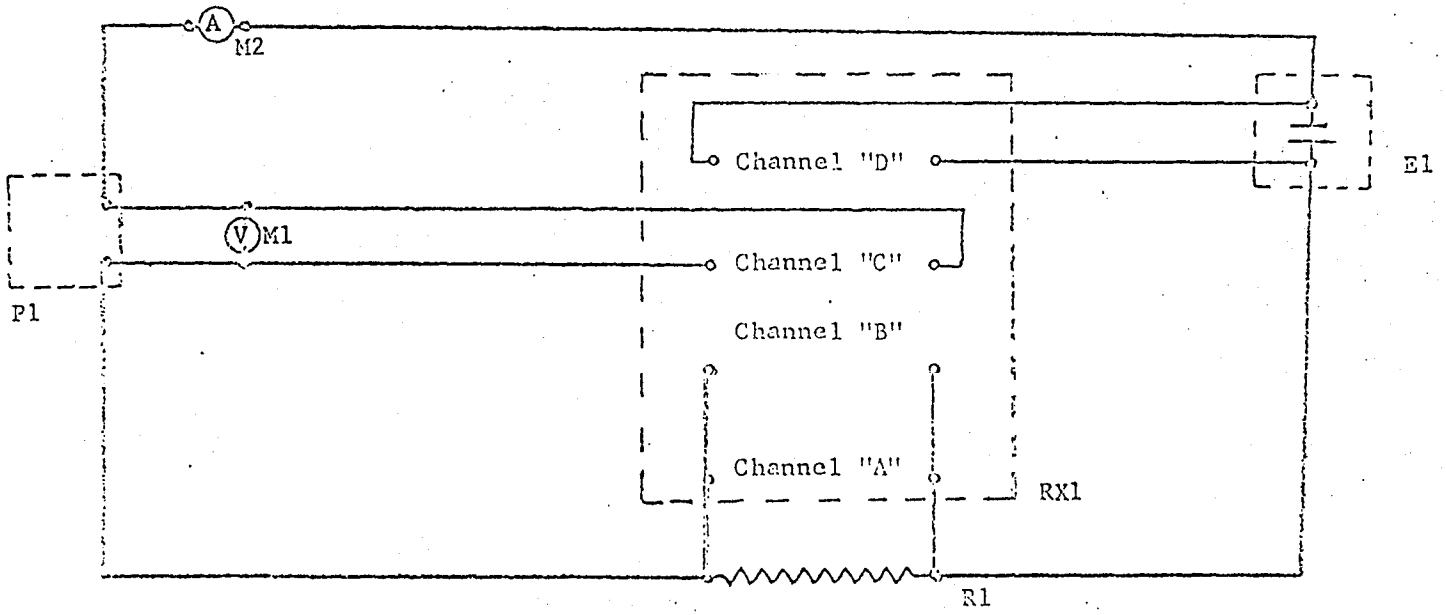
HEAT PAPER RESISTIVITY CIRCUIT - Figure 7




P1 - Regatran P/S Model PV36-30-11
(setting 20 volts; 20 amps. max.)
M1 - Voltmeter; D.C. Weston Model No. 622
(accuracy ± 0.5%)
M2 - Ammeter; Weston Model No. 901
(accuracy ± 0.3%)
R1 - Resistor; 100 ohms
RX1- Recorder; Sandborn D.C. Series 150
Channel "A" - calibrated to 0 - 40 ma
Channel "B" - calibrated to 0 - 400 ma
Channel "C" - 0 to 20 volts
Channel "D" - 0 to 20 volts
E1 - Electrode Plates

[redacted]

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