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MEMORANDUM REPORT ARBRL-MR-03322

CHARACTERIZATION OF MAPLE CHARCOAL USED TO MAKE BLACK POWDER

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November 1983

US ARMY ARMAMENT RESEARCH AND DEVELOPMENT CENTER
BALLISTIC RESEARCH LABORATORY
ABERDEEN PROVING GROUND, MARYLAND

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The chemical composition and physical properties of charcoal, used in the manufacture of black powder, was determined. Data include elemental composition, true density, surface area, particle size distribution and volatile content. It was concluded that different samples of charcoal from a particular "lot" differ in composition. The suggestion was made that to produce a more predictable black powder, the charcoal will have to be more uniform and will require some preblending.

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I. INTRODUCTION

The relationship between volatile content of charcoal to the burning rate of black powder has been discussed by several authors,[1,2,3,4] and such comments have been summarized in two Ballistic Research Laboratory, BRL, reports.[5,6] The conclusion from these works is that a charcoal with a 25% volatile content is the optimum concentration to achieve the fastest burning black powder. This volatile level has been part of an informal specification for charcoal used in black powder since 1930. The subject of charcoal and its volatile content is particularly germane now as the Army Ammunition Plant, AAP, at Charlestown, IN, produced its first black powder that will be evaluated for its combustion and ballistic properties. Additionally, Eli Freedman,[7] of the BRL, will perform thermodynamic calculations to predict maximum pressure, impetus, adiabatic flame temperature, and the molecular weight of product gases using the actual chemical composition of the ingredients. Because of these investigations, it would seem desirable to correlate performance properties to the raw materials employed, particularly to the physical and chemical properties of charcoal. Aside from the question of adequacy, which will be evaluated by a gun firing program sponsored by DARCOM, another concern was to characterize charcoal as fully as possible such that the same black powder product could be produced again. This principle can be extended by considering different lots or different types of charcoal and, in these cases, one would like to form a data base from which to make comparisons to quantify both differences and similarities.

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[1] *A. Kirshenbaum, Thermochimica Acta, Vol. 18, p. 113, 1977.*

[2] *W. Hintze, Explosivstoffe, Vol. 2, p. 41, 1968.*

[3] *E. Gray, H. March and J. Robertson, "The Influence of Charcoal in the Combustion of Black Powder," RARDE, Fort Halstead, Sevenoaks, England. Presented at Basic and Applied Pyrotechnic's International Conference, ARCHCHON, France, October 1982.*

[4] *J.E. Rose, "Investigation on Black Powder and Charcoal," IHTR-433, Naval Ordnance Station, Indian Head, MD, September 1975.*

[5] *R.A. Sasse', "The Influence of Physical Properties on Black Powder Combustion," ARBRL-TR-02308, Ballistic Research Laboratory, USA ARRADCOM, Aberdeen Proving Ground, MD, March 1981 (AD A100273).*

[6] *R.A. Sasse', "Strand Burn Rates of Black Powder to One Hundred Atmospheres," ARBRL-TR-02490, Ballistic Research Laboratory, USA ARRADCOM, Aberdeen Proving Ground, MD May 1983 (AD A129087).*

[7] *E. Freedman, to be published in Proceedings of 20th JANNAF Combustion Meeting, October 1983.*

II. THE CHARACTERIZATION OF CHARCOAL

Black powder was fabricated by AAP using maple charcoal supplied in 40-pound bags by the Roseville Charcoal Co., of Zanesville, OH. Fifty bags were on each pallet and 50 such pallets were purchased. Five pallets were sampled where two bags were opened and a one-pound portion was scooped from each. Our brevity in sampling reflects the erroneous opinion that the samples would be much alike. Chemical and physical analyses were performed on these samples as well as on oak and maple charcoals used in the "deviant lot" series of black powders.[8] In fact, these latter charcoals were used to prepare laboratory samples that were subject to extensive study at the BRL.[5,6]

Results are given in Table 1 where percentages are by weight and it can be seen that the chemical composition and physical properties varied among the samples. Two samples taken from the same container are in reasonable agreement as are the data for two samples taken from the same pallet. It would be reasonable to assume that the two bags sampled were on the top layer and the bags were probably close to one another.

Of particular interest is the range in volatile content, 21 to 29 percent, measured from the thermogravimetric, TGA, curves. These extremes in weight loss are shown in Figure 1 where the curves were obtained by first evacuating the sample and measuring the weight loss as a function of temperature, to 950° C, in a stream of argon. Near 375° C some samples lost more weight than did others and in these cases, a prominence appeared. Curves of this type are labeled B in Table 1, whereas curves with a smoother shape are labeled A. This effect becomes evident when examining the entire TGA data set as given in Figures 2-12. The maple and oak TGA curves for the charcoals used in the "deviant lot" series of black powder are compared in Figure 12. One would expect the differences noted in the TGA data are related to the slower burn rate of the oak black powder as compared to maple. One additonal experiment was conducted where dry maple wood was converted to charcoal in the TGA apparatus; the data are given in Figure 13. The derivative is also shown in this figure, and two peaks can be observed indicating two different depolymerization reactions. The steepness of the curve exemplifies careful temperature control is required to produce a charcoal with a particular volatile content. Gray, et al,[3] expressed a similar concern regarding the steepness of their carbon and hydrogen data depicting the roasting of Alder wood.

The measurement of volatiles conforms to the standard test described; however, the measured weight loss is not just due to the simple vaporization of material. Heating charcoal is a depolymerization reaction that breaks chemical bonds; in some cases, new structures are formed making nonvolatile materials as well as fragmented structures that will vaporize. The measurement of weight loss upon heating embraces both of these effects and is termed "volatile content."

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[8] *J.C. Allen, "Scope of Work for MM & TE Project 5764303, Acceptance of Continuously Produced Black Powder," Report No. SARPA-QA-X-10, Picatinny Arsenal, Dover, NJ, November 1975.*

Other variances were noted in the data given in Table 1, particularly ash content where values ranged from two to ten percent. Values were measured by the standard way of heating charcoal in air to 950°C and the residual weight was taken as various oxides; however, the cations exist as compounds other than the oxides in the original charcoal. Under these circumstances the values in Table 1 cannot be accurately adjusted to represent properties of the ash-free state. In this data set, converting the values to an ash-free basis would not render the various quantities as constants.

The range of ash content seemed large and one would like to know if this variance were associated with a different type of tree chemistry. It was hoped that elemental chemical analysis of the ash would show trends if, in fact, ash were related to other properties. The ash was dissolved and calcium, sodium, potassium, lithium, iron, and magnesium were analyzed by atomic absorption techniques. Silicon, aluminum, titanium, and phosphorous were determined by spectrophotometric techniques. Analysis of the ash, charcoal elemental analysis, and energy content of charcoal was performed by the Fuel and Engineering Company of Thornwood, NY using appropriate ASTM test methods and certification with samples from National Bureau of Standards is maintained. In examining the elemental content of ash in Table 1, no distinct trend between composition and other properties was found. Further, it appeared that high ash samples had much of the same proportionate composition as did the low ash material. One obvious effect of high ash content is that such charcoals contain less combustibles.

The true density was measured by Robin J. Davala of TBD using a helium pycnometer, and values ranged between 1.44 to 1.56 gm/cm^3. Sample six was heated to 950°C in flowing argon for 35 minutes, and the resulting carbon had a density of 1.77 gm/cm^3. Correlation between density and carbon or volatiles is not supported by the data set.

Analysis for carbon and hydrogen was performed combusting charcoal with oxygen in a tube furnace and passing carbon dioxide and water through absorbents and measuring them gravimetrically. Nitrogen was determined by the Kjeldahl method and sulfur was analyzed gravimetrically as barium sulfate. The amount of oxygen was taken as the difference between 100% and the sum of the constituents found in charcoal.

The total carbon content of the samples ranged from 71 to 80%, but the variance does not relate to volatile content. Although the values vary, the empirical formula for charcoal is near $C_8 H_4 O$ which could represent an extensive fused ring system.

The heat of combustion was measured in an adiabatic calorimeter, and the values in Table 1 ranged over 13%.

Two other observations deserve comment. The first surprise was the charcoal samples had markedly different size distributions and charcoals containing small particles were of high volatile content. In effect, the high volatile material appeared more frangible. The second surprise was the very low surface areas that were measured by James Haberman of LCE in Dover, NJ, using the B.E.T. technique and it would appear that most of the pores in charcoal are plugged. Of the limited number of samples measured, the ones with the higher surface area are more dense.

III. SUMMARY

Various chemical and physical analyses were performed in an attempt to: (1) characterize charcoal used in black powder and (2) to form a data set that could be used to compare future charcoal samples. From the various analyses, it was shown that charcoal properties can vary among a group of samples purchased from the same supplier representing the same "lot." The variations noted probably reflect the individual properties of the wood employed and the temperature histories in different locations in the kiln. It would seem that to make a more predictable black powder, the charcoal will have to be more uniform, requiring preblending.

TABLE 1. CHARACTERIZATION OF ROSEVILLE MAPLE CHARCOAL[1]

Sample Number	Density g/cm³	Volatiles %	TGA Type	Chemical Characteristics[2]							Size		
				O_2 %	C %	H_2 %	N_2 %	S %	Ash %	Btu lb	>100 Mesh %	100-200 Mesh %	<200 Mesh %
1	1.45	23.1	A	12.71	78.40	3.26	0.44	0.08	5.11	12,744	46.82	38.06	15.12
		23.5		12.87	78.39	3.22	0.43	0.09	5.00	12,782			
2	1.47	23.5	A	13.10	78.40	3.24	0.35	0.01	4.90	12,614	46.98	36.61	16.42
		23.1		13.25	78.50	3.19	0.33	0.01	4.72	12,649			
3	1.50	24.1	A	14.20	75.83	3.15	0.35	0.02	6.45	12,267	46.84	35.96	17.20
		22.4		13.75	76.07	3.11	0.35	0.01	6.71	12,289			
4	1.51	21.9	A	14.14	75.41	3.24	0.34	0.02	6.85	12,194	42.99	35.14	21.88
		23.7		14.14	75.46	3.30	0.36	0.02	6.72	12,234			
5	1.56	22.1	A	15.27	71.96	2.06	0.46	0.03	10.22	11,463	20.21	24.83	54.96
		21.0		14.13	72.09	2.94	0.46	0.03	10.35	11,436			
6	1.54	24.6	A	14.25	72.93	2.84	0.48	0.02	9.48	11,665	32.54	33.76	33.70
		24.8		14.47	72.74	2.84	0.47	0.02	9.46	11,658			
7	1.45	27.0	B	14.18	79.44	3.39	0.32	0.02	2.65	12,873	27.35	13.54	59.11
		27.5		14.22	79.15	3.46	0.32	0.01	2.84	12,846			
8	1.44	28.0	B	14.47	79.25	3.44	0.30	0.01	2.53	12,836	29.34	14.33	56.33
		28.4		14.36	79.16	3.52	0.30	0.01	2.65	12,853			
9	1.48	26.4	B	15.39	77.48	3.26	0.33	0.01	3.53	12,691	15.60	11.17	73.23
		29.5		15.01	77.72	3.29	0.33	0.01	3.64	12,599			
10	1.45	28.4	B	16.03	76.87	3.49	0.32	0.01	3.30	12,539	36.61	18.19	45.20
		29.0		15.98	76.91	3.38	0.30	0.01	3.42	12,540			
Maple	1.47	23.7	B-	12.69	80.95	2.96	0.30	0.01	3.09	12,962			
				12.62	80.94	2.92	0.30	0.02	3.20	12,975			
Oak	1.63	22.0	A	13.25	70.59	2.92	0.61	0.04	13.05	11,007			
				12.69	70.82	2.65	0.62	0.06	13.15	11,029			
6 Carbonized	1.77												

TABLE 1. CHARACTERIZATION OF ROSEVILLE MAPLE CHARCOAL (CONT'D)

	Ash Composition[2]											Surface Properties[3]	
Sample Number	SiO_2 %	CaO %	Al_2O_3 %	K_2O %	Na_2O %	Li_2O PPM	Fe_2O_3 %	TiO_2 %	MgO %	P_2O_5 %	Recovered %	Area m^2/g	Pores cm^3/g
1	34.28	28.44	13.97	9.66	2.45	1163	4.58	0.34	2.33	1.26	97.31	2.22	2
3	42.63	30.05	12.66	12.93	1.64	1040	2.55	0.31	2.22	1.15	106.14	4.72	4
5	25.97	44.55	13.71	9.58	1.00	456	2.27	0.28	1.87	1.28	100.51	15.74	10
7	16.82	43.32	14.04	12.85	1.72	1161	2.79	0.33	3.68	2.05	97.60	3.27	3
9	16.01	46.50	13.09	8.60	1.82	1332	4.85	0.22	3.19	1.72	96.00	3.65	5
Maple	18.02	44.91	12.37	8.37	1.60	1214	2.54	0.28	3.42	2.13	93.64	3.05	3
Oak	22.88	44.91	12.20	6.01	0.79	396	1.78	0.22	1.56	0.92	91.27	17.61	15

Comments: Sample number; two 40 lb. bags of charcoal sampled per pallet, each pallet holding a total of 50 bags. In this characterization, five pallets were sampled from the 30 available, i.e., 1-2, 3-4, 5-6, ... Two analyses were performed on each bag of charcoal chosen for study as shown by double entries in the table.

Density; helium pycnometer values. Analysis performed by Robin J. Davala, DRDAR-TBD, ARRADCOM, Aberdeen, MD.

Volatiles; weight loss as sample is heated from 150 to 950°C at 20°C/minute in flowing argon. Values relate to dry sample.

TGA Type: two different curvatures noted in TGA data; see graphs.

Chemical[2] and Physical Composition; values relate to dry sample and present chemical composition includes ash. Samples include volatiles. Oxygen calculated by difference.

Btu/lb; weight includes ash and volatiles, but not moisture.

Carbonized; sample heated to 950°C in argon for 35 minutes.

Oak and Maple Entries, samples from "deviant lot series" procured for the MM&TE project, "Acceptance of Continuously Produced Black Powder". Oak purchased from Hardwood Charcoal Company.

[1]Roseville Charcoal Used in the Army Ammunition Prove Out Program, 1983.

[2]Analysis performed by Fuel Engineering Company of NY.

[3]Analysis performed by J. Haberman, DRDAR-LCE-C, ARRADCOM, Dover, NJ.

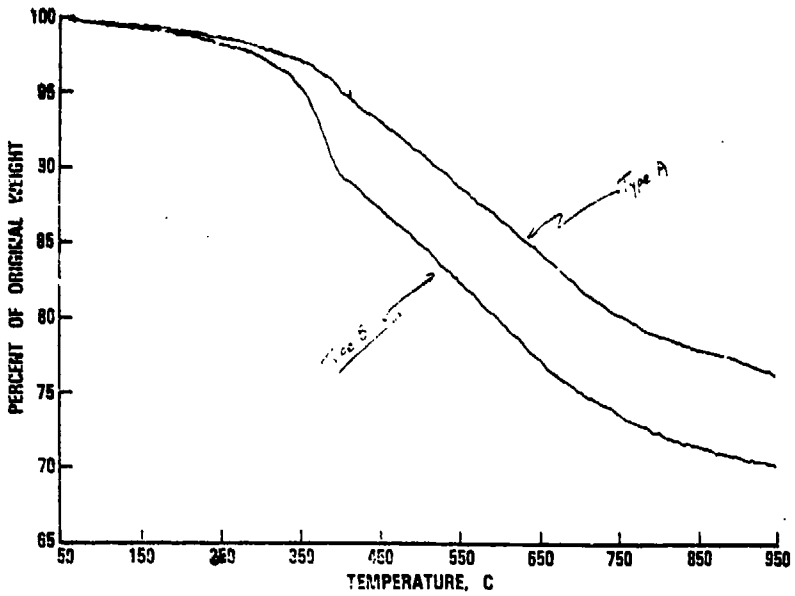


Figure 1. TGA Data Showing Low Volatile Sample; Sample Number 10 - Type A Compared To High Volatile Sample; Sample Number 1 - Type B.

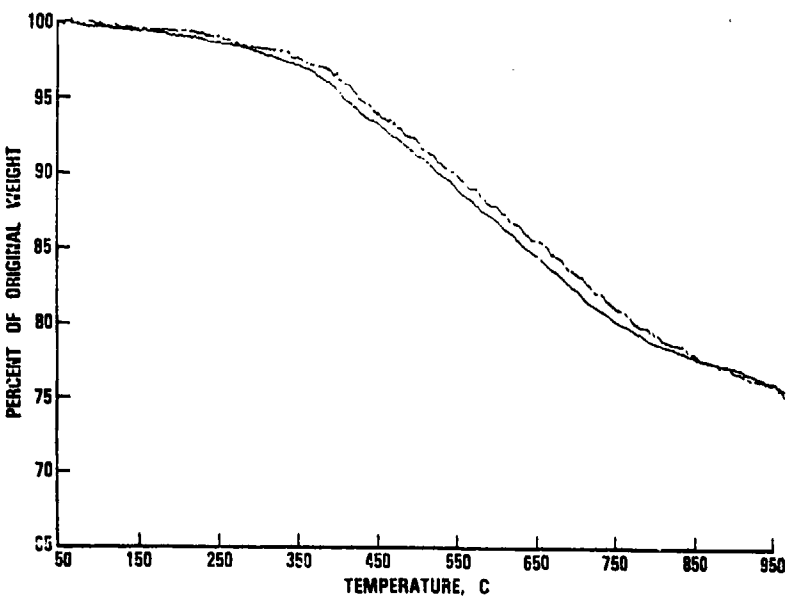


Figure 2. TGA Data; Sample Number 1.

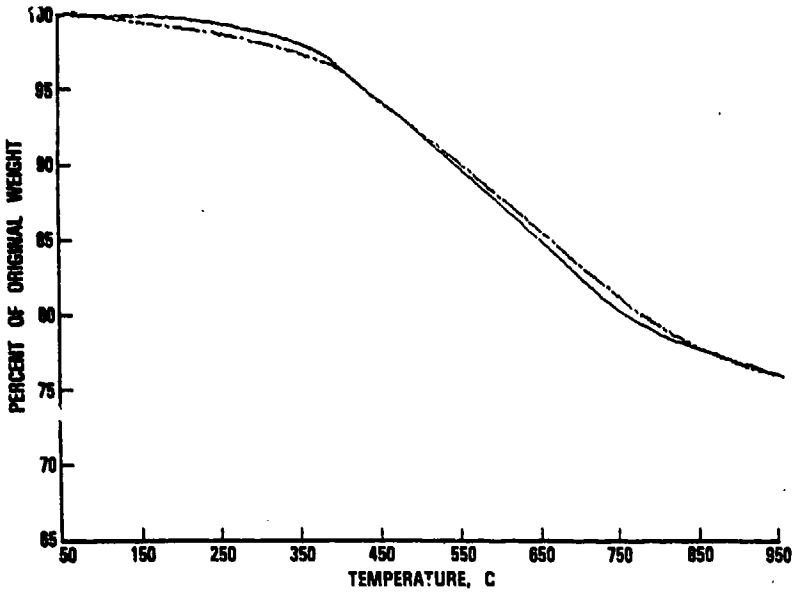


Figure 3. TGA Data; Sample Number 2.

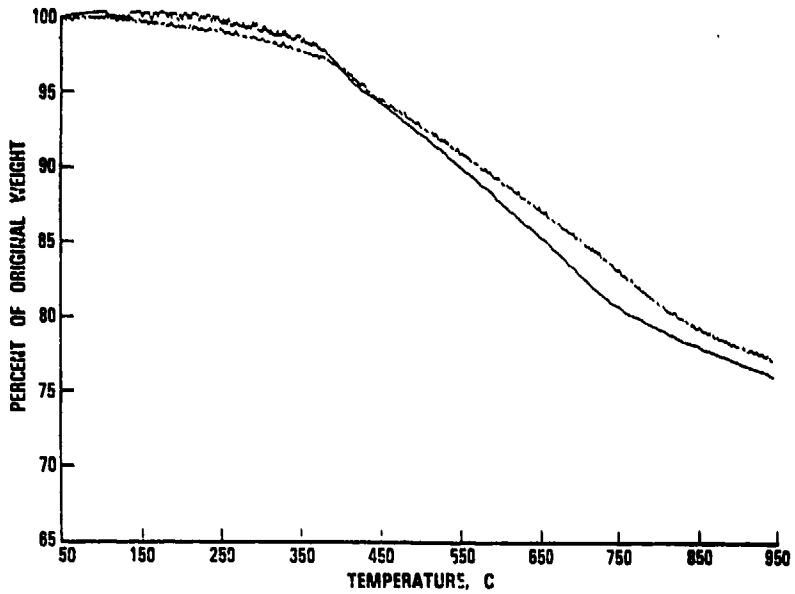


Figure 4. TGA Data; Sample Number 3.

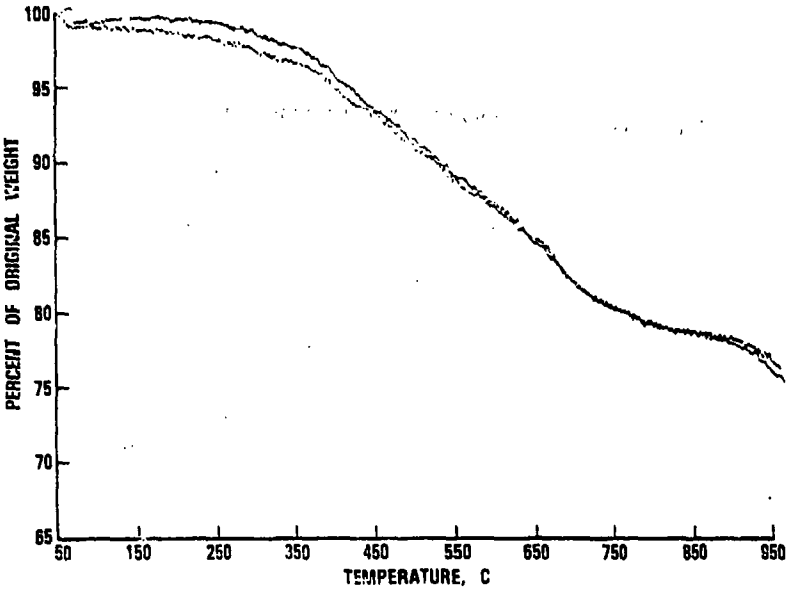


Figure 5. TGA Data; Sample Number 4.

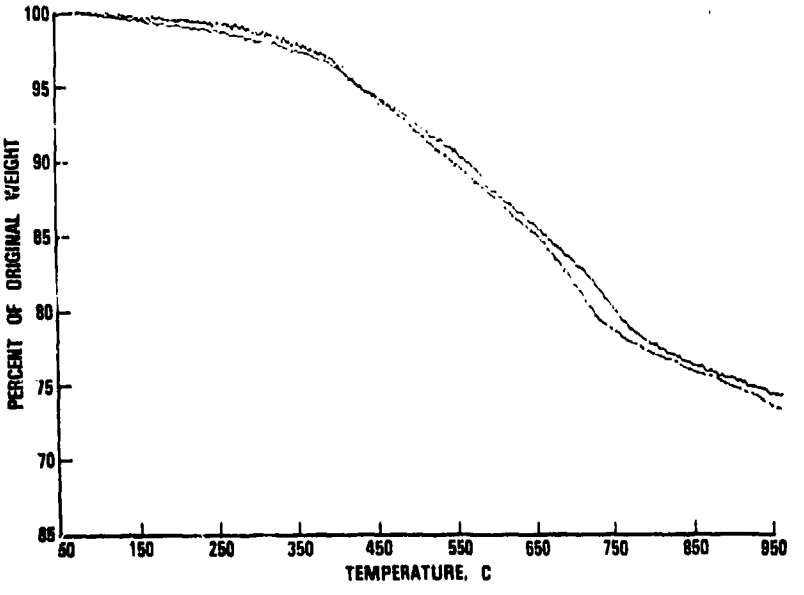


Figure 6. TGA Data; Sample Number 5.

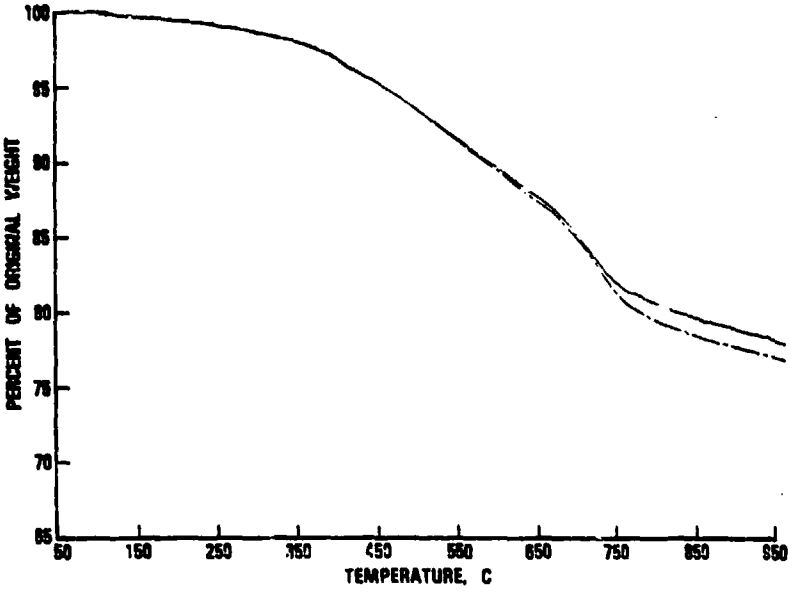
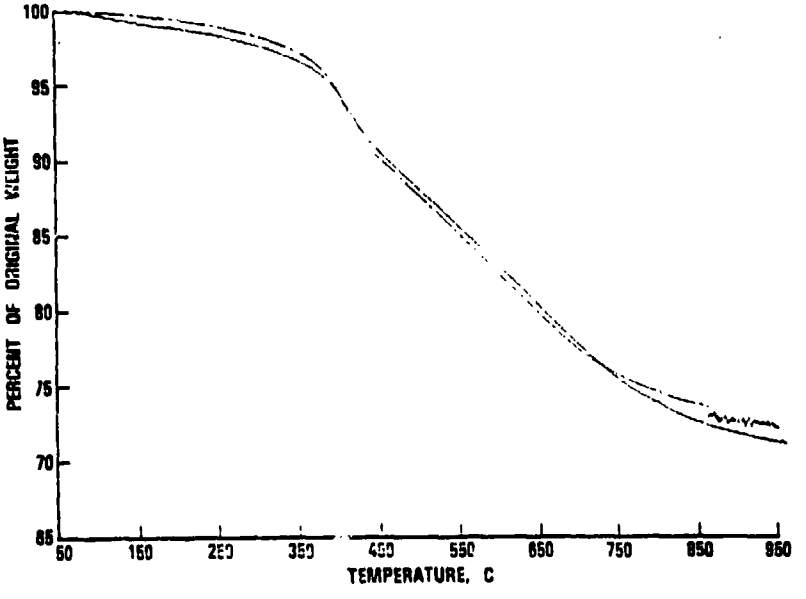


Figure 7. TGA Data; Sample Number 6.



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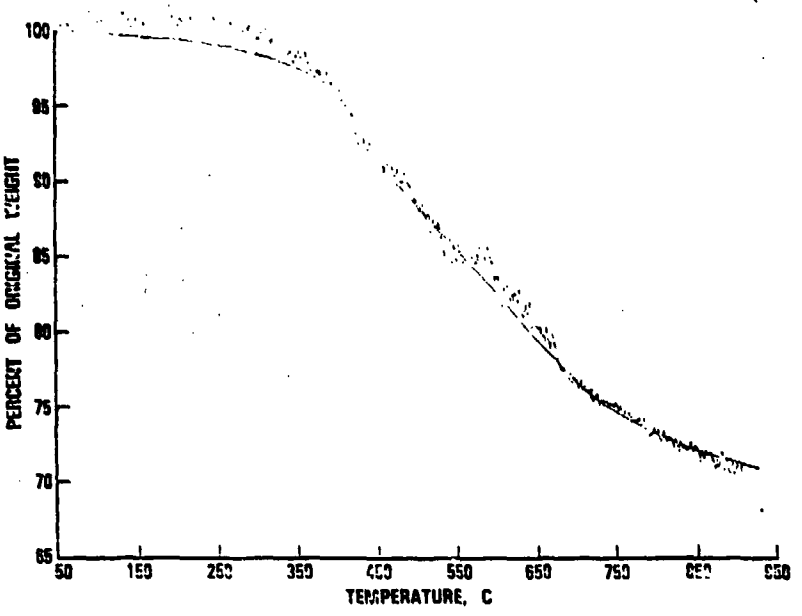

Figure 9. TGA Data; Sample Number 8.

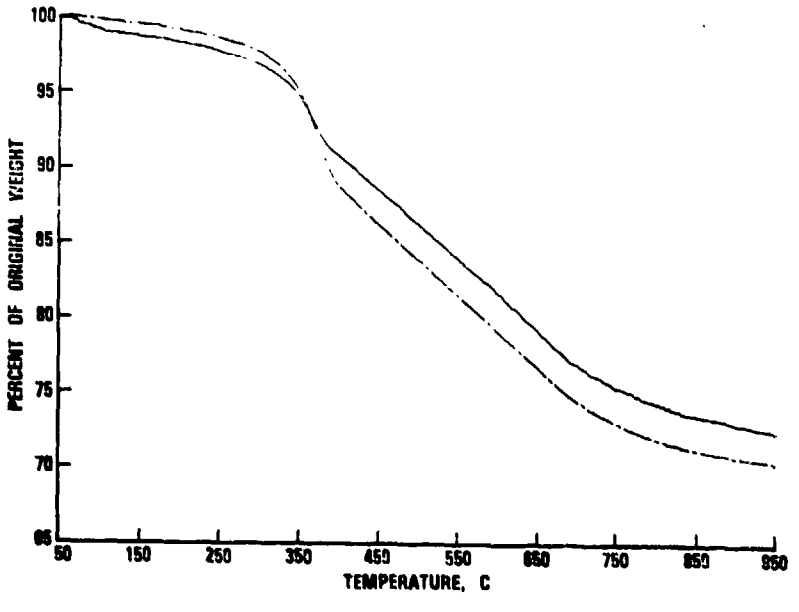

Figure 10. TGA Data; Sample Number 9.

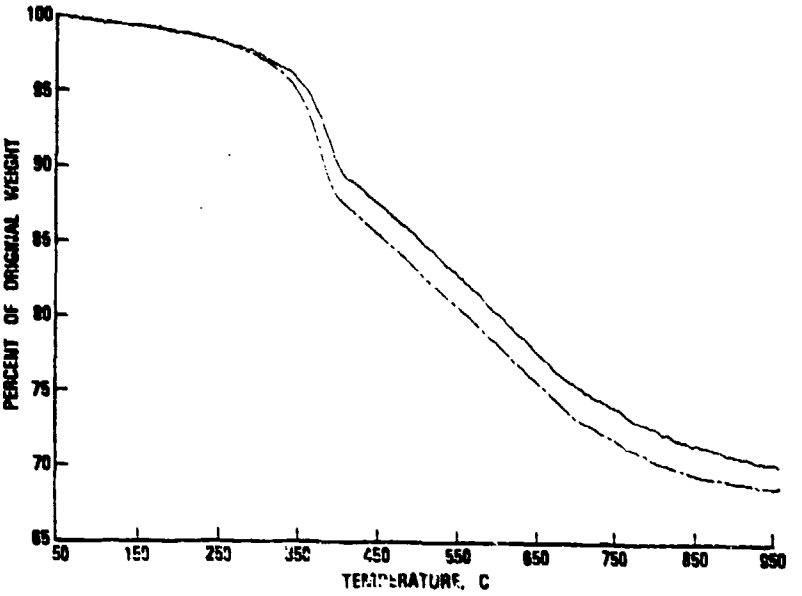

Figure 11. TGA Data; Sample Number 10.

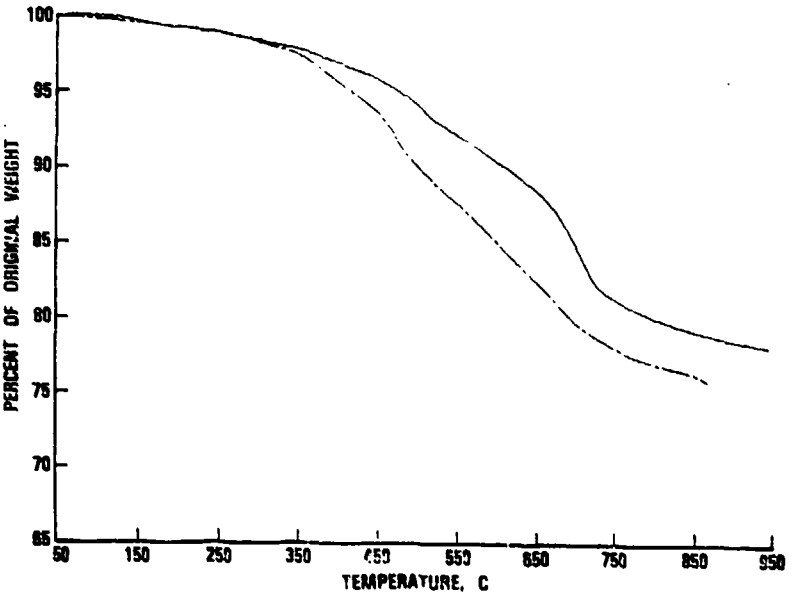

Figure 12. TGA Data Of Oak And Maple Charcoals Used In The "Deviant Lot" Series; Solid Line Oak And Dashed Line Maple Charcoal.

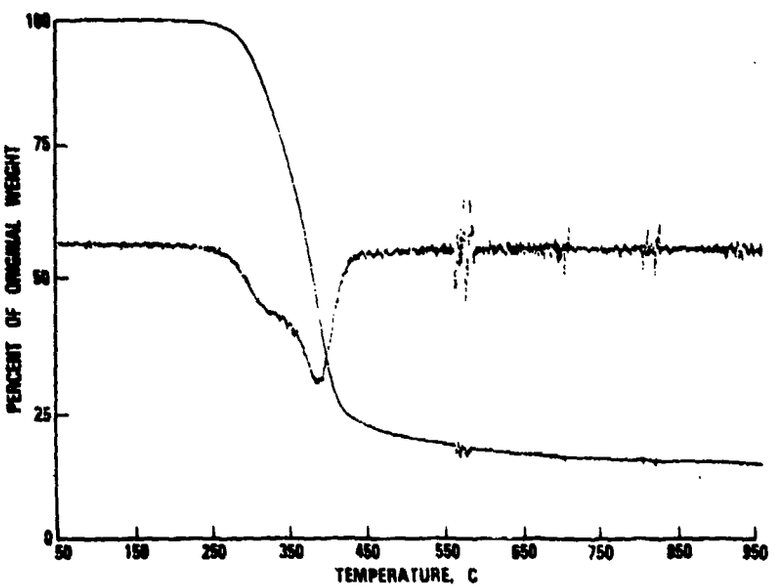

Figure 13. Solid Line TGA Data For Maple Wood And The Dashed Line Is Its Derivative.

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7. E. Freedman, to be published in Proceedings of 20th JANNAF Combustion Meeting, October 1983.

8. J.C. Allen, "Scope of Work for MM & TE Project 5764303, Acceptance of Continuously Produced Black Powder," Report No. SARPA-QA-X-10, Picatinny Arsenal, Dover, NJ, November 1975.

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