

THE JOURNAL

OF THE

AMERICAN CHEMICAL SOCIETY.

THE COMPOSITION OF CERTAIN MODERN POWDERS.

BY CHARLES E. MUNROE.

THE powders examined belong principally to the class known in commerce as nitro-powders, and they are of interest since it is claimed for them that, when used as ballistic agents, they are more or less smokeless.

The method of analysis employed was as follows: The substance was first dried to constant weight over calcium chloride and the loss thus determined is noted as "volatile," though it was principally, when not wholly, hygroscopic moisture. A portion of the well-dried sample was then digested in ether-alcohol until disintegrated, the viscous solution formed was somewhat diluted and then poured upon an asbestos filter.

These filters were prepared by drawing out very light glass test tubes to a narrow funnel and chocking the neck with asbestos, which was exhausted with ether-alcohol and ether, and dried to constant weight. The filter with its charge was now suspended in a beaker partly filled with ether-alcohol so that the powder was immersed in the liquid, the whole covered so as to prevent loss by volatilization, and when the viscous fluid had passed through, the residue was washed with ether-alcohol until solution ceased. The residue was then dried to constant weight. This immersion was found necessary to prevent clogging by evaporation.

The ether-alcohol filtrate was treated with three volumes of chloroform to precipitate the soluble cellulose nitrates, which

were then collected on a filter, dried, and weighed. They appear in the tables under the technical name of "nitro-cotton." The filtrate from the ether-alcohol-chloroform solution was evaporated and the salts present determined. The residue insoluble in ether-alcohol was treated with boiling water so long as any solvent action continued. The residue then remaining was again dried and weighed. Finally it was exhausted with ethyl acetate and again dried and weighed. The loss by exhaustion with ethyl acetate is noted as "gun cotton" and the residue as cellulose. The metallic salts present were now determined in the aqueous solution.

When nitroglycerine was found to be present the dried powder was first exhausted with ether in a Soxhlet extractor and the loss noted as nitroglycerine, though as shown later it sometimes included resins or oil which were present in slight proportion.

When the powder was readily attacked by water, as was the case with the American Wood and Brackett Powders, the aqueous treatment preceded that by ether-alcohol, and the salts were thus extracted together with the product of the charring of the wood which is called "humus" in the tables. This "humus" was determined by evaporating the aqueous extract to constant weight on the water bath, then treating with nitric acid repeatedly until oxidation ceased, again evaporating to dryness, and then fusing gently.

To determine the aurin, which appears in several of the powders the sample was exhausted with chloroform in a Soxhlet extractor, and the residue in the tared flask weighed. The residue was then taken up with a few drops of chloroform, transferred to a separating funnel, shaken with strong ammonia water, and separated. The chloroform solution was repeatedly washed with dilute ammonia until no pink color was developed in the water, then the chloroform solution was again evaporated in the tared flask and the weight of the residue determined. The difference in the two weights is the aurin.

The results obtained were as follows :

Schultze Gunpowder.—The powder was marked "Improved Waterproof, New Issue," and was in the form of nearly spherical

grains which were almost white in color, though having a slight yellowish cast, and of such a size as to pass through a No. 20 mesh sieve. The results of the analysis on the specimen as received were as follows :

	Per cent.
Nitro-cotton.....	27.71
Gun cotton.....	32.66
Cellulose.....	1.63
Paraffin	4.20
Barium nitrate	27.62
Sodium nitrate	2.88
Potassium nitrate	2.47
Volatile	1.48
	<hr/>
	100.65

E. C. Gunpowder.—The package was marked E. C. Smokeless Sporting Powder, No. 1. "1 lb. of the Powder is equivalent to 2 lbs. of the Best Black Powder. This powder does not absorb moisture from the atmosphere; but should it be accidentally wetted it may be again rendered fit for use by drying at a temperature not exceeding that of boiling water, and afterwards exposing it to the air for a day or more."

The grains were similar in size and shape to those of the Schultze powder described above, but they were orange colored. The composition was found to be :

	Per cent.
Nitro-cotton.....	53.57
Gun cotton.....	1.86
Cellulose.....	3.12
Barium nitrate	34.26
Sodium nitrate	3.67
Potassium nitrate	1.48
Aurin	0.55
Volatile	1.17
	<hr/>
	99.68

American Wood Powder, Grade C.—This powder was in rounded grains, of a dark brown color, and of such size as to pass through a No. 21 mesh. According to the accompanying circular Grade C "is for general use. Medium coarse. Good in any gun. Is strong, gives good pattern, and is the best all-

round powder made." The composition was found to be as follows :

	Per cent.
Soluble nitro-lignin	29.25
Insoluble nitro-lignin	14.06
Lignin (charred)	28.08
Humus.....	10.32
Sodium nitrate	15.27
Volatile	3.01
	<hr/>
	99.99

American Wood Powder, Grade E.—This powder resembled Grade C in form and color of grain, but passed through a No. 32 mesh. The accompanying circular stated that this grade is "Fine. The quickest of the lettered grades. For 16 to 20 bore guns, and rifles smaller than 45 calibres."

Analysis gave the following :

	Per cent.
Soluble nitro-lignin	24.91
Insoluble nitro-lignin	25.62
Lignin (charred)	19.15
Humus.....	8.65
Sodium nitrate	17.81
Volatile	3.86
	<hr/>
	100.00

American Wood Powder. Ten Bore Trap.—This powder had a brown color and was in rounded grains of such size as to pass through a No. 19 mesh. According to the circular this grade "is about of size of C. Is too quick for larger bores."

Analysis gave :

	Per cent.
Soluble nitro-lignin	33.21
Insoluble nitro-lignin	18.69
Lignin (charred)	20.27
Humus.....	10.65
Sodium nitrate	14.82
Volatile	2.36
	<hr/>
	100.00

American Wood Powder—Twelve Bore Trap.—This powder had a brown color and was in rounded grains of such size as to pass through a No. 24 mesh. The circular stated that "Twelve bore trap is very quick. In size about the same as D. It is too quick for 10 bores unless very small loads are used."

Analysis gave :

	Per cent.
Soluble nitro-lignin	29.47
Insoluble nitro-lignin	21.85
Lignin (charred)	16.59
Humus.....	15.62
Sodium nitrate	13.38
Volatile	3.14
	<hr/> 100.05

American Wood Powder.—The foregoing samples were all purchased of D. Kirkwood, of Elm street, Boston, Mass. Another sample obtained from Martin and Company, of Newport without any mark, consisted of light brown rounded grains about the size of fuze powder, which were highly porous and quite friable.

Analysis gave :

	Per cent.
Soluble nitro-lignin	24.90
Insoluble nitro-lignin.....	30.07
Lignin (lightly charred).....	19.55
Humus.....	9.89
Sodium nitrate	9.76
Volatile	5.83
	<hr/> 100.00

Brackett's Sporting Powder.—The sample of this powder which was also obtained from D. Kirkwood, Elm street, Boston, Mass., had the same general appearance as the American Wood Powders except that its grains were larger. About one-half of these grains passed through a twenty mesh sieve, while the remainder passed through a twelve mesh sieve. The circular stated that this powder was a strong quick powder intended for wing and bush shooting. Like the American Wood Powders the grains were porous and friable.

Analysis gave :

	Per cent.
Soluble nitro-lignin	31.43
Insoluble nitro-lignin	13.70
Lignin (charred)	13.22
Humus.....	18.94
Sodium nitrate	19.76
Volatile	2.93
	<hr/> 99.98

S. K. Powder.—This package was marked "Smokeless (S.K.) Rook Rifle Gun Powder, made by the Smokeless Powder Company, Limited, at Barwick Works, Herts, England." The grains resembled those of the E. C. powder described above, except that the S. K. grains were more nearly lenticular and were pink colored. The granulation was such that the powder all passed through No. 24 sieve, and the granulation was quite uniform. The powder emitted a slight fragrant odor suggesting purified wood spirits.

Analysis gave :

	Per cent.
Nitro-cotton.....	20.39
Gun cotton.....	57.73
Aurin (alkaline)	1.11
Barium nitrate	18.08
Potassium nitrate	1.24
Volatile.....	1.43
	<hr/> 99.98

It will be noted that the coloring matter found, aurin, is the same as that found in the E. C. powder, yet the S. K. powder is colored pink or purple red while the E. C. was orange colored. As aurin (pararosolic acid, $C_{19}H_{14}O_3$) is orange colored when neutral, and pink to purple red when treated by alkalies, it is evident that the varying conditions of neutrality accounts for the difference in color.

The powder appeared to have been waterproofed and as, when the chloroform solution from the powder was just passing to dryness, a faint odor of nitrobenzene was discerned, it is possible that this agent, together with wood spirits was used superficially to produce this effect. The waterproofing was not, however, very efficient, for, after three hours immersion in cold water, the grains were found to be very much swollen, while the water was colored yellow, possessed the characteristic odor of the powder, and deposited aurin and nitrates on evaporation.

S. R. Powder.—This sample was marked "Smokeless (S. R.) Rifle Gunpowder made by the Smokeless Powder Company, Limited, at Barwick Works, Herts, England." This powder resembled the S. K. powder described above in color, odor, and

form of grain, except that the S. R. passed through No. 16 sieve (though much of it was smaller) and that the odor was very strong.

Analysis gave :

	Per cent.
Nitro-cotton.....	28.18
Gun cotton.....	46.97
Aurin (alkaline)	1.06
Barium nitrate	19.97
Potassium nitrate	2.35
Volatile	1.45
	<hr/> 99.98

As with the S. K. powder these grains were disintegrated by immersion in cold water.

Rifleite Gunpowder.—This powder, which was manufactured by the Smokeless Powder Company, Limited, at Barwick Works, Herts, England, was described on the package as being made for use with the Magazine Rifle 303. It was in the form of nearly square flat grains having the color and luster of graphite and the odor of wood spirits, which had apparently been used in waterproofing the grains. This waterproofing was not, however, completely efficacious, since when the powder was first immersed in cold water a considerable portion of the graphite floated off and rose to the surface of the water, and when the moist powder was subsequently dried in a porcelain dish, the graphite adhered to the walls of the dish. When the graphite, which was a wholly superficial covering, was removed from the grains, they were found to be faintly yellow colored and translucent. The water used in this operation had acquired the odor of the powder and on evaporation to dryness it left a yellow residue.

The powder offered considerable resistance to the action of ether-alcohol, but the grains gradually swelled up, split into laminæ and were eventually completely disintegrated after seventy-two hours exposure to ether-alcohol in the cold.

The coloring matter, which was quite soluble in ethyl alcohol, was rather insoluble in ether-alcohol, and on the addition of ether to the alcohol solution this coloring matter was precipitated in beautiful golden yellow plates, which were identified as a phenyl-

amidoazobenzene, $C_6H_5.N_2.C_6H_4NH$ (C_6H_5), the sulphonate of which is known in commerce as Manchester Yellow.

Analysis gave :

	Per cent.
Nitro-cotton.....	22.48
Gun cotton.....	74.16
Phenyl amidoazobenzene	2.52
Graphite	Trace
Volatile	0.84
Total.....	100.00

The grains were found to average 0.0638 inch in length, 0.0612 in width, and 0.0155 in thickness, though occasional ones were nearly double this length.

I am indebted to the courtesy of the New England representative of the U. S. Cartridge Company for these samples of powder from the Barwick Works.

Maxim Powder.—I am indebted to the courtesy of Dr. J. E. Blömen, chemist to the Forcite Powder Company for two samples of this powder. The powder, which was evidently intended for use for small arm charges, was in two forms, one rectangular flat grains, and the other in short cords. The color was dark brown, the flat grains being somewhat translucent, while the cords were opaque. Both possessed a distinct fatty odor, but this was less marked and may have been accidental in the flat grains, as, when received, a few of the rods had become mixed with this sample. The flat grains were quite tough and offered considerable resistance to fracture, but the cord had very slight cohesive properties and fractured on application of slight force, like half dried putty.

A mean of the measurements of ten of the flat grains gave, length 0.0512 inch, width 0.459 inch, thickness 0.0166 inch.

Analysis gave :

	Per cent
Nitro-cotton.....	8.14
Gun cotton.....	71.19
Sodium carbonate	2.58
Nitroglycerine	17.90
Volatile	0.19
	100.00

The average of the measurements of ten of the cord formed grains gave, length 0.25 inch, diameter 0.0456 inch. The ends of these grains were rough as if they had been broken from the rods, so the micrometer measurement for the lengths is only approximate.

Analysis gave :

	Per cent.
Nitro-cotton	6.84
Gun cotton.....	46.60
Nitroglycerine	44.60
Sodium carbonate	1.70
Volatile	0.26
	<hr/>
	100.00

The odor of castor oil was plainly distinguishable even in the cold, and when the powder was immersed in cold water for as short a time as three hours, the water became yellow colored and yielded a fatty residue on evaporation, but we have no good method as yet for separating this oil or resin quantitatively from nitroglycerine.

German Smokeless Powder.—This sample was received with the statement that it was a German powder procured from Cologne for trial in the 8" B. L. Rifle. The powder was in the form of nearly cubical grains about 1 cc. in volume and averaging 1.6203 grams each in weight. The surface had the luster and color of graphite and the cubes appear as if they had been cut from a larger mass with a saw. The grains were of such consistency that they could just be indented with the finger-nail. On cutting the grains, the graphitic appearance was found to be wholly superficial, the interior having the appearance of explosive gelatine, though having a greater consistency than the latter substance.

Analysis gave :

	Per cent.
Nitro-cotton.....	48.83
Gun cotton.....	7.45
Nitroglycerine	43.15
Graphite	Trace
Volatile	0.53
	<hr/>
	99.96

I am deeply indebted to Mr. G. W. Patterson for his valuable assistance in this work.