

# Speculation on the Explosive Decomposition of “Yellow Powder”

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## Introduction

Attending a short-course in pyrotechnic chemistry provided the opportunity for the authors to study a little-understood historic explosive currently viewed primarily as a novelty and chemical curiosity. Part of the course was the preparation of a seminar. The instructors proposed the “yellow powder” seminar topic for the authors.

Yellow powder is a combination of potassium nitrate, potassium carbonate and sulfur. Alone, the combination of potassium nitrate ( $\text{KNO}_3$ ) and sulfur (S) burns with difficulty. Potassium carbonate ( $\text{K}_2\text{CO}_3$ ) is used as the main component in some dry chemical fire extinguishers. However, a melted and slightly cooked mixture of three parts  $\text{KNO}_3$ , two parts  $\text{K}_2\text{CO}_3$  and one part S (sometimes called “yellow powder”) can react with great violence. The purpose of the following communication is to relate the authors’ experiences with “yellow powder”. An extensive on-line search was performed to supplement some information supplied by the instructors. Some (very limited) experimentation and additional research followed the initial literature research. Nonetheless, the course instructors suggested that the results of this brief study should be shared by preparing this article.

## Historical Overview

Following are the principal historical developments of this seventeenth-century novelty labeled “yellow powder”:

- 1) “Yellow powder” behaves very similar to *aurum fulminans* (fulminating gold) and *aurum tonitruans* (thundering gold), well-known explosive substances.<sup>[1]</sup>
- 2) In at least two historical accounts of experiences with “yellow powder”, reference was made to placing the reactants on an iron surface (plate, bowl, shovel).<sup>[2,3]</sup> We were quite curious about the possible role of the iron surface on the chemistry. This curiosity will be elaborated in this paper.
- 3) The formation of “yellow powder” and its subsequent explosive nature was stated to be dependent upon proper heating. Rapid heating tended to result in the combustion of sulfur—forming  $\text{SO}_2$ —and the melting and eventual decomposition of potassium nitrate. Under-heating results in the slow sublimation and dissipation of the reactants eliminating the potential of any explosive reaction. Johann Rudolf Glauber,<sup>[4]</sup> a seventeenth century chemist, designed an apparatus for properly heating the reactants needed to make “yellow powder”. He also elaborated on the need for proper heating to produce the characteristic explosion.
- 4) In the eighteenth century Antoine Baumé proposed and showed that potassium polysulfide is an intermediate in the reaction by first reacting sulfur with potassium carbonate to produce potassium polysulfide. He also proposed that the precursor to the explosion was “nitrous sulfur”.<sup>[5]</sup>

## Guiding Questions

Upon considering the historical development of this seventeenth century novelty, five important questions were formulated to help guide our efforts. Prior to addressing the questions, a sample of “yellow powder” was prepared by mixing 0.30 g of potassium nitrate, 0.20 g of anhydrous potassium carbonate, and 0.10 g of powdered sulfur together with a wooden stir-stick. Approximately 0.1 g of this mixture was

placed on a metal spatula and heated gently with a propane torch. After several minutes, the evolution of a gas was noted, and the powder began to acquire a golden-brown color. Shortly after the gas evolution ceased, there was a rapid (1–2 seconds) change in color to a deeper shade of brown/gold. The mixture then exploded leaving virtually no residue.

### Guiding Questions for this Study

- 1) Do polysulfides really form as intermediates?
- 2) Would the reasonably stable polysulfide intermediate explode when mixed with potassium nitrate and then properly heated?
- 3) Does an iron heating surface play a significant role in the chemistry?
- 4) Is “nitrous sulfur” the precursor to the explosion?
- 5) Can a sequence of equations be formulated to describe the process?

## Experimental Results and Discussion

### Do the Polysulfides Really Form as Intermediates?

In an attempt to produce one or more polysulfides, a mixture of potassium carbonate and sulfur were reacted; 0.10 g of sulfur and 0.20 g of anhydrous potassium carbonate were mixed together. Approximately 0.1 g of this mixture was placed on a metal spatula and heated gently with a propane torch. During the heating process, the melted mixture bubbled (probably  $\text{CO}_2$  and trace amounts of other side reaction gases were produced) then the material turned reddish brown and crumbly when cooled. Upon referencing the *Merck Index*<sup>[6]</sup> and the *CRC Handbook of Chemistry and Physics*,<sup>[7]</sup> it seems most likely that the reddish brown solids were a mixture of  $\text{K}_2\text{S}_4$  and  $\text{K}_2\text{S}_5$ . Thus, it was concluded that polysulfides form when potassium carbonate is heated with sulfur.

### Would the Reasonably Stable Polysulfide Intermediates Explode When Mixed with Potassium Nitrate and Then Heated?

To 0.30 g of the previously prepared potassium polysulfide powder, 0.30 g of potassium nitrate was added and mixed. Approximately 0.1 g of this “yellow powder” mixture was placed on a metal spatula and heated gently with a propane torch. After melting and a brief period of continued heating of the polysulfide-potassium nitrate product, an explosion occurred. This further gives credence to the speculation that polysulfide intermediates were produced and that they are quite capable of producing an explosion when reacted with potassium nitrate. Subsequent literature research<sup>[8,9]</sup> supported the conclusion regarding the production of potassium polysulfide intermediates from the reaction of potassium carbonate with sulfur.

### Does an Iron Heating Surface Play a Significant Role in the Chemistry?

A properly proportioned mixture of potassium carbonate, sulfur, and potassium nitrate was heated in a porcelain crucible behind a protective shield to determine if iron played a role. Upon heating, the reactants exploded. Thus it was concluded that iron does not play a significant role in the reaction chemistry.

### Is “Nitrous Sulfur” the Precursor to the Explosion?

In responding to this question the following was also considered. Is the original reaction between potassium carbonate, sulfur, and potassium nitrate a combustion reaction using potassium nitrate as an oxidizer, or is the explosive reaction occurring via an alternate reaction? To answer this question it was proposed that potassium perchlorate be used as a potential oxidizer rather than potassium nitrate. (Note that in pyrotechnics potassium perchlorate is generally considered to be a more vigorous and powerful oxidizer than potassium nitrate.) When potassium perchlorate was substituted for potassium nitrate in a similar stoichiometric amount, a rapid combustion ensued, but there was no explosion. Accordingly, it was concluded that the nitrogen in the potassium nitrate probably does play an important role in the reaction.

Based on Baumé's suggestion of "nitrous sulfur" as an intermediate,<sup>[5]</sup> a preliminary search was conducted to identify sulfur nitride compounds that may be explosive and bear similar physical properties to the observed reaction intermediate. Initial descriptions found in the *Merck Index*<sup>[6]</sup> and the *CRC Handbook of Chemistry and Physics*<sup>[7]</sup> led us to conclude that the reaction most likely produced tetrasulfur tetranitride (S<sub>4</sub>N<sub>4</sub>) and disulfur dinitride (S<sub>2</sub>N<sub>2</sub>). This suspicion was further confirmed when more detailed descriptions of these compounds were located in several sources described below.

The first formally published synthesis and characterization of tetrasulfur tetranitride was done by Becke-Goehring<sup>[10]</sup> in 1960 utilizing sulfur(II) chloride and ammonia. Follow-up work by Villena-Blanco and Jolly<sup>[11]</sup> in 1967 provided additional information and observations regarding the properties of tetrasulfur tetranitride. Villena-Blanco and Jolly report that tetrasulfur tetranitride, which is "golden poppy" colored, is explosive and that the "sensitivity of S<sub>4</sub>N<sub>4</sub> toward both shock and temperature increases with its purity".

Cotton and Wilkinson<sup>[12]</sup> describe tetrasulfur tetranitride as forming thermochromic crystals that range from orange-yellow at 25 °C to red above 100 °C. Tetrasulfur tetranitride has a square-planar molecular geometry containing alternating sulfur and nitrogen atoms. This highly stressed geometry could help account for its shock sensitivity and explosive nature. Cotton and Wilkinson caution that the compound must be handled with care since "grinding, percussion, friction, or rapid heating can cause it to explode". These statements are consistent with our observations during the induction period immediately preceding the explosion of "yellow powder".

Disulfur dinitride is also reported<sup>[12]</sup> to be stable in air, but is potentially explosive in response to shock, friction or heat, especially. Disulfur dinitride has a square-planar molecular geometry containing alternating sulfur and nitrogen atoms. Similar to tetrasulfur tetranitride, this highly stressed geometry helps account for its shock sensitivity and explosive nature.<sup>[13,14]</sup> Recent Raman and X-ray studies of tetrasulfur tetranitride and disulfur dinitride confirm the square-planar geometries, as well as the physi-

cal and chemical properties noted in the sources mentioned earlier.<sup>[8,14]</sup>

### Can a Sequence of Equations Be Formulated To Describe the Process?

The **unbalanced** proposed equations are as follows:

1. Production of potassium polysulfides



2. Production of nitrous sulfides



3. Explosion

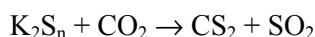


The above sequence was the consensus view of the authors; however, the following sequence was also considered as a possible alternative:

1. Production of potassium polysulfides



2. Production of carbon disulfide



3. Production of nitrous sulfides



4. Explosion



The above progression indicates a somewhat different involvement of the carbonate ion.

### Conclusion

Due to the limited duration of the short course and very limited lab facilities, questions and instrumental confirmation of these proposed reactions remain speculations. The capture and analysis of the exit gases during the gentle heating, as well as after the explosion, may confirm the proposed reactions. Mass spectrometry, Raman spectroscopy, and <sup>15</sup>N NMR spectroscopy could help in distinguishing the components. An exhaustive search of the Chemical Abstracts Services database for articles pertaining to the explosive decomposition of the sulfur nitrides may also provide an insight to the reaction pathways suggested here.

It is hoped that this brief communication may spur additional interest in this phenomenon and potential clarification of the events that take place during the gentle heating of “yellow powder”.

### References

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