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Research paper

Radiative Properties of a Red Phosphorus Based Combustion Flame

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Abstract: The spectral energy, E_λ (in $\text{J}\cdot\text{g}^{-1}\cdot\text{sr}^{-1}$), of a model obscurant composition containing red phosphorus (P(red), 59 wt.%), Zr (15 wt.%), potassium nitrate (KNO_3 , 21 wt.%) and a polyacrylate binder (5 wt.%), was determined in band II ($\lambda = 3\text{-}5\ \mu\text{m}$) and band V ($\lambda = 8\text{-}14\ \mu\text{m}$). The spectral energy obtained in band V was higher than the spectral energy in band II. The band ratio V/II and spectral energy indicated that the combustion flame of the pyrolant cannot be described as a graybody. A combined DTA/TG analysis under anaerobic conditions revealed an initial exotherm followed by an endothermic process, the latter coinciding with a mass loss of >40 wt.%, indicative of the vaporization of P(red).

Keywords: combustion flames, infrared radiation, red phosphorus, obscurant, phosphorus pentoxide, radiometry

Symbols and Abbreviations

Band II 3.0-5.0 μm range

Band V 8-14 μm range

DTA Differential thermal analysis

E_λ Spectral energy at wavelength (λ) [$\text{J}\cdot\text{g}^{-1}\cdot\text{sr}^{-1}$]

MTV	Magnesium, Teflon [®] , Viton [®] , (standard blackbody infrared flare composition)
m_{res}	Residual mass [g]
P(red)	Red phosphorus
T_{ad}	Adiabatic combustion temperature [K]
TMD	Theoretical maximum density [$\text{g}\cdot\text{cm}^{-3}$]
TGA	Thermogravimetric analysis
A	Oxygen balance [wt.% oxygen]
λ	Wavelength [μm]
$\Theta_{\text{II/V}}$	Band II to band V ratio [–]
ζ	Mass fraction [wt.%]

1 Introduction

Pyrolants containing high mass fractions of P(red) ($\zeta > 50$ wt.%) serve to produce smoke in naval location markers [1] and multispectral obscurants [2]. In these formulations, the exothermicity of a primary reaction between a supplementary fuel and an oxidizer yields sufficient energy to volatilize the P to give both P_4 and P_2 , which then burn in a diffusion flame with the atmospheric oxygen to yield phosphorus pentoxide (P_2O_5). If access to air is insufficient, phosphorus trioxide (P_2O_3) is formed as the major product. These oxides at high temperatures exist in an equilibrium with P_4O_6 , P_4O_{10} , PO_2 , PO , P and O_2 [3]. The initial hydrolysis of these species with atmospheric moisture yields phosphinic acid (H_3PO_2), diphosphoric acid ($\text{H}_4\text{P}_2\text{O}_7$) and other open chain-polyphosphoric acids ($\text{H}_{2n}\text{P}_n\text{O}_{3n+1}$), which all react consecutively with atmospheric moisture to finally yield orthophosphoric acid, the orthophosphoric acid then hydrates further [4, 5].

While the obscuration properties of these formulations are well reported [6-10], the radiative properties of their combustion flames are not. It is therefore the objective of the present investigation to determine the radiative properties of the combustion flames of a model obscurant formulation containing P(red).

2 Materials

2.1 Safety note

P(red) and Zr are substances that are extremely sensitive to electrostatic discharge. Therefore, all manipulations must be conducted with

electrically conductive and appropriately earthed equipment. As both P(red) and Zr also pose an extreme danger of fire, flameproof personal protective equipment, such as NOMEX™, PROBANTM or comparable qualities with balaclava and face shield and protective gloves must be worn at all times when handling these substances. Personnel handling these substances must wear electrically conductive shoes and an earthing wristband at all times. The final pyrotechnic formulations and the transient mixtures are also highly sensitive towards electrostatic discharge, friction and impact and must be handled accordingly. Though these formulations serve as slow burning obscurants under confinement, these formulations have been observed to transition to detonation and may yield a catastrophic response when present in larger amounts.

2.2 Tested materials

Pyrotechnic compositions were prepared in the following composition ratios and from the following ingredients:

- P(red) (HB 801, Clariant): 59 wt.%,
- potassium nitrate (KNO_3 , $\geq 99\%$, TROPAG): 15 wt.%,
- Zr (Grade GH, Chemetall): 21 wt.%,
 - (i) Zr + Hf > 98 wt.%; Hf ~ 2 wt.%, H 0.8 ± 0.2 wt.%,
 - (ii) grain size iaw Blaine: 5.5 ± 1 μm , $> 99.9\%$ passes 325 mesh sieve,
 - (iii) rim value combustion time: 460 ± 75 s for 50 cm;
- polyacrylate binder: 5 wt.%.
Polyacrylate, Hytemp® 4454 (Zeon), was used for the formation of the polyacrylate binder. Ethyl acetate was $\geq 99.5\%$, Ph EUR (Carl Roth). An oxygen underbalanced formulation ($A = -88.86$ wt.%) was derived from the disclosure in [11].

The formulation was prepared on a 1 kg batch size in a 5 L stainless steel planetary blender with a proprietary constructed water powered heating jacket, (Herbst Maschinenfabrik GmbH) equipped with a circumferential scraper from conductive polymer (Kalrez®).

P(red) was added to the blending bowl, which was connected to a thermostat and kept at 25 °C and wetted sufficiently with ethyl acetate to yield a pasty consistency after a few revolutions. The KNO_3 , which had initially been passed through a 50 μm sieve, was then added and more solvent was added to preserve the pasty consistency of the mix.

In a separate stainless steel bowl, Hytemp® chunks were dissolved overnight in ethyl acetate with some agitation from a magnetic stirring bar on an IKA magnetic laboratory blender. The Zr powder was added to the slightly opaque

lacquer and was mixed thoroughly. The magnetic stirring bar was removed and rinsed with solvent to remove residual Zr and lacquer.

The Zr lacquer dispersion was poured onto the top of the solvent moist P(red), KNO_3 mix, and remaining lacquer was rinsed in with more ethyl acetate and scraped off from the bowl with an electrically conductive elastic spoon. The mix was then blended under safe conditions for 20 min with the heating jacket kept at 25 °C. An exhaust system carried away the emanating solvent fumes. At the end of the mixing period the composition appeared dark brown and had a crumbly morphology. It was removed from the mixing bowl and spread out as a layer (≤ 2.54 cm (1 inch) thick) on craft paper placed on a stainless steel sieve rack that was transferred to a ventilated heating cabinet supplied with warm air (30 °C, 50%RH).

After drying overnight (≥ 12 h), the formulation was separated into 40 g batches prior to pressing. The formulations were kept in the same cabinet until ready for consolidation. The consolidation was achieved in a Dunks hydraulic press, at 20 tons using a cylindrical pressing mold (internal diameter 40 mm) with electropolished surfaces. A total of 22 pellets were pressed and tested from a 1 kg batch.

3 Test Methods

3.1 Radiometric tests

The cylindrical pellets were placed on a heat resistant tile made of fire clay between stainless steel splint pins. Ignition was facilitated by a T15-electric igniter enhanced with a quickmatch fixed with adhesive tape on top of the pellets. The pellets were burned in a radiometric tunnel with a suction fan ($3 \text{ m}\cdot\text{s}^{-1}$ air speed) removing the aerosol to the back of the pellet.

Upon airstream in the test tunnel the pellets yielded an orange yellow flame with dimensions ~ 15 cm (height) by ~ 15 cm (width). The radiometric performance was determined in two bands using band pass filters, for band II: (1.9-3.0 μm), and band V: (8-14 μm), with an IR radiometric system (RM 6600 and an uncooled pyroelectric detector RkP 575, all of Laser Probe, USA). The radiometer had been calibrated with a blackbody (SR-32, CI Electro-Optical Systems, Haifa, Israel) at $T = 1000$ K.

The radiometric readings (in $\text{W}\cdot\text{sr}^{-1}$) in both channels were separately integrated and the results of 22 measurements were used to determine the mean values.

3.2 Thermoanalytical investigation

A combined thermogravimetric and differential thermal analysis was carried out in a NETZSCH STA 449C. The purging rate was 150 mL/min Ar 5.9. The sample mass was 5 mg, the crucible was made from sintered Al_2O_3 .

3.3 Chemical analysis of the combustion residues

Chemical analysis was conducted at Umweltlabor Westpfalz GmbH (Kaiserslautern, Germany). The sample of the burnt material was digested with aqua regia. The soluble fraction was analyzed with atomic absorption spectrometry (AAS) and flame emission spectrometry (FES). P was determined as molybdatovanadatophosphate. The insoluble fraction showed the presence of both a major amount of Zr and a minor amount of P (<5 wt.%) by means of X-ray fluorescence. No attempt was made to quantify these contents. The pellets were weighed both after pressing and after combustion. Out of 22 pellets, 5 disintegrated upon combustion/removal from the firing holder and were hence not measured.

3.4 Chemical equilibrium calculation

A chemical equilibrium calculation at constant pressure was carried out with EkviCalc [12].

4 Results and Discussion

P(red)-based obscurant formulations require a heat source based on a fuel/oxidizer mixture to effect volatilization of P(red) [2]. Many fuel/oxidizer combinations are feasible (Mg/PTFE, Mg/ KNO_3 , ZrNi/ CsNO_3 , B,Si/ KNO_3 , *etc.*) and have been used in qualified ammunitions. However, not all of these are useful from the standpoint of investigating the properties of a P-based combustion flame, as both volatile (SiO [13], BO_2 [14]) or dispersed combustion products from a diffusion flame ($\text{C}_{(\text{graphite})}$ [15], SiO_2 [16]) could influence the radiative properties of the flame. Hence it was decided to study a system applying a secondary fuel that neither yields a diffusion flame to avoid ejection of condensed particles nor produces any volatile emitters. Therefore, Zr was chosen as the fuel as it does not satisfy Glassman's criterion for vapour phase combustion [17]. Oxidizer KNO_3 was selected, as Zr/ KNO_3 pyrolants show little combustion flame and burn virtually pressure insensitively [18]. Although Zr/ KNO_3 react to form zirconium dioxide (ZrO_2), which has a high emissivity in the infrared [19], this will probably not be ejected with the P vapour and hence will not affect the combustion

flame emissivity. Furthermore, the products of possible side reactions, ZrP [20] and K_3PO_4 , which may form in the primary reaction, are all refractory in nature and hence are unlikely to be dispersed into the flame, but rather sinter together.

The combined DTA and TG analysis of the formulation (Figure 1) indicated two major processes. An exothermic reaction with onset at 309 and peak 370 °C coincides with a mass loss of ~3 wt.%. In view of the thermal investigations reported in [18], it is reasonable to assign this to the combustion reaction between KNO_3 and Zr releasing nitrogen, which accounts for about 2% of the weight loss. An endotherm peak with an onset at 417 and peak 452 °C is associated with a mass loss of about 56%, which almost fits the amount of P(red) in the formulation.

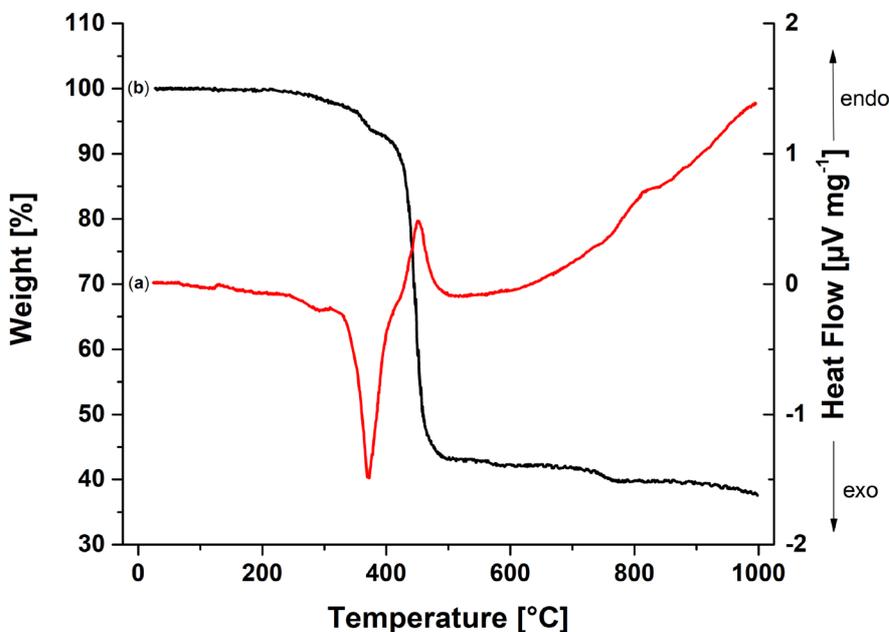
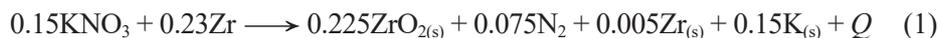


Figure 1. Combined DTA (a) and TGA (b) of the formulation under Ar with high purge rate ($150 \text{ mL}\cdot\text{min}^{-1}$)

The weighing of the burnt pellets revealed a similar mass loss of 60%, being in good agreement with the DSC/TGA results. Chemical analysis of the combustion residue gave a P content of 3% in the part of the sample that dissolved in aqua regia (~34 wt.%), while the remainder, being insoluble in aqua regia, showed minor amounts (<5 wt.%) of P by X-ray fluorescence, which however cannot be determined exactly. Assuming an overall mass fraction of 4% P in the

combustion residue ($m_{\text{res}} = 16.8$ g), the actual mass of P released amounts to 23 g, which corresponds to 97.5 wt.% of the P originally contained in the formulation.

It is therefore assumed that the combustion starts in the condensed phase with a reaction between Zr and KNO_3 . This reaction yields ZrO_2 , K and nitrogen which accounts for the first decrease in mass.



The heat of reaction facilitates vaporization of the P (Equation 2), which on contact with air burns to form P_4O_{10} (Equation 3).



In view of the residual P and K content in the burnt pellets (Table 1) it can be assumed that some KNO_3 reacts with P to give K_3PO_4 (see Table 4).

Table 1. Chemical composition of the burnt pellets based on dry mass

Element	Content [wt.%]
K	8.5
P	3

The combustion process of consolidated P rich formulations is peculiar, in that the pellets do not show a receding burning surface area. Instead, the approximate geometrical dimensions of a pellet are retained during combustion, with only a slight expansion along the pressing axis taking place. This is because P impedes the hydrocarbon combustion by quenching radicals and trapping oxygenated species [21], leaving mainly a porous carbon-rich residue. Thus, the porous binder matrix mainly serves as a diffusion aid and flame holder for the burning P vapour.

Table 2 lists the mean spectral energy, E_{λ} , of the formulation in band II and band V determined from the instrumented burns in a flare test tunnel.

Table 2. Radiometric properties of RP-based pyrolant consolidated at 25 tons (76% TMD)

Parameter	Unit	Value
Experimental density (% TMD)	$\text{g}\cdot\text{cm}^{-3}$	1.9 (76)
TMD	$\text{g}\cdot\text{cm}^{-3}$	2.488
$E_{\text{band II}}$	$\text{J}\cdot\text{g}\cdot\text{sr}^{-1}$	51.0 ± 2.7
$E_{\text{band V}}$	$\text{J}\cdot\text{g}\cdot\text{sr}^{-1}$	65.0 ± 3.5
$\Theta_{\text{V/II}}$	–	1.27

While there is plenty of data in the public domain on the spectral energy in band II (the present value compares nicely with second generation spectral flare formulations [22]), there is very little information to be found about band V spectral energies for flare formulations. Yayla [23] systematically investigated the influence of various additives on the spectral energy of MTV (59/27/14) in three wavebands. Yayla reported spectral energies in a band a little narrower than the present band V ($\lambda = 8\text{-}10 \mu\text{m} = 1.9 \text{ J}\cdot\text{g}^{-1}\cdot\text{sr}^{-1}$) and the corresponding band II energy ($78 \text{ J}\cdot\text{g}^{-1}\cdot\text{sr}^{-1}$) [23].

The intensity ratio between two bands of a black- or gray-body radiator can be used to derive its temperature [24]. While P(red)-based flames cannot be described as a graybody in the visible [25], it has been reported that they emit graybody radiation in the infrared range [26]. A corresponding determination was therefore attempted. For a range of temperatures, the radiance ($\text{W}\cdot\text{m}^{-2}\cdot\text{sr}^{-1}$) in band II and band V and the corresponding ratio $\Theta_{\text{C/B}}$ were calculated with Planck's law [27] (see Figure 2 and Table 3).

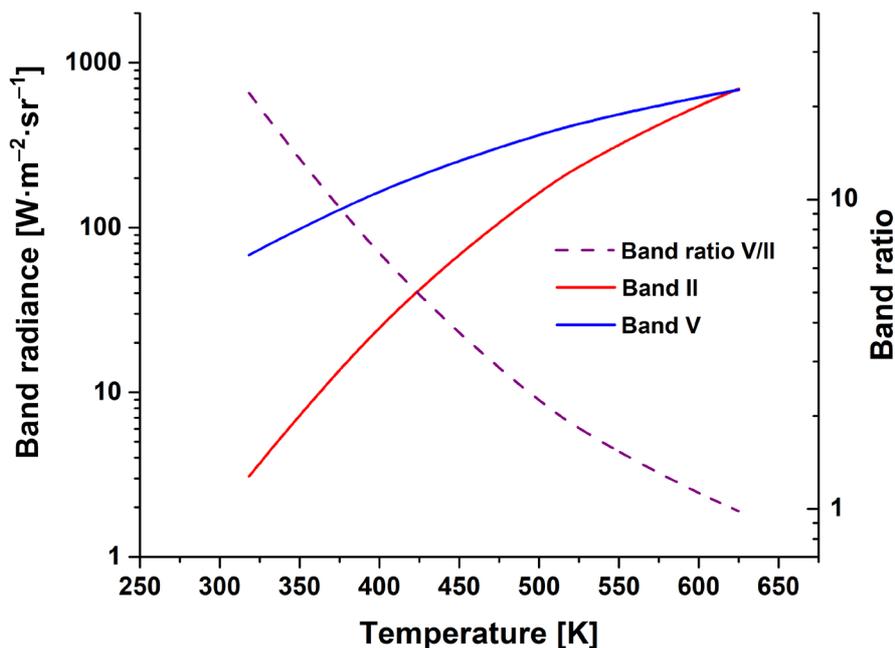


Figure 2. Radiance for a blackbody ($\varepsilon = 1.0$) in band II (3-5 μm) and band V (8-14 μm) and the ratio between the bands as a function of its temperature

Table 3. Radiance and band ratio for a blackbody at $T = 580$ K

Parameter	Unit	Value
Temperature	K	580
Band II (3-5 μm)	$\text{W}\cdot\text{sr}^{-1}\cdot\text{m}^{-2}$	445.2
Band V (8-14 μm)	$\text{W}\cdot\text{sr}^{-1}\cdot\text{m}^{-2}$	563.5
$\Theta_{\text{V/II}}$	–	1.27

The observed band ratio, $\Theta_{\text{V/II}}$, fits an ideal blackbody radiator at $T = 580$ K. Without doubt, this temperature is far too low to account for the actual combustion temperature.

The calculated adiabatic combustion temperature (T_{ad}) (Figure 3) for the pyrolant itself is already much higher ($T_{\text{ad}} = 1366$ K), and the diffusion flame of the primary combustion products ($\text{P}_{4(\text{g})}$, $\text{P}_{2(\text{g})}$, $\text{PN}_{(\text{g})}$, CO , H_2 , *etc.*) yields between $T_{\text{ad}} = 1400$ -2400 K depending on the amount of air entrained into the primary combustion plume (Table 4). This is in good accord with experimental findings by Koch [28, 29], who reported combustion flame temperatures

for a P(red) rich incendiary B299 ($P_{\text{(red)}}/Mg = 75/25$) of 1473 K. Table 4 lists the major species (residue 0.0001) calculated with the EkviCalc code under isobaric conditions (pressure 0.1 MPa).

Table 4. Chemical compositions

Condensed species		Gaseous species	
Species	Mass fraction	Species	Mass fraction
ZrO _{2(s)}	0.2026	P _{4(g)}	0.3555
K ₃ PO _{4(s)}	0.1468	P _{2(g)}	0.1304
C _(graphite)	0.0160	H _{2(g)}	0.0038
		PN _(g)	0.0826
		CO	0.0164
		CHP _(g)	0.0243
		N ₂	0.0072
		HCP _(g)	0.0049
		P ₄ O _{6(g)}	0.0094

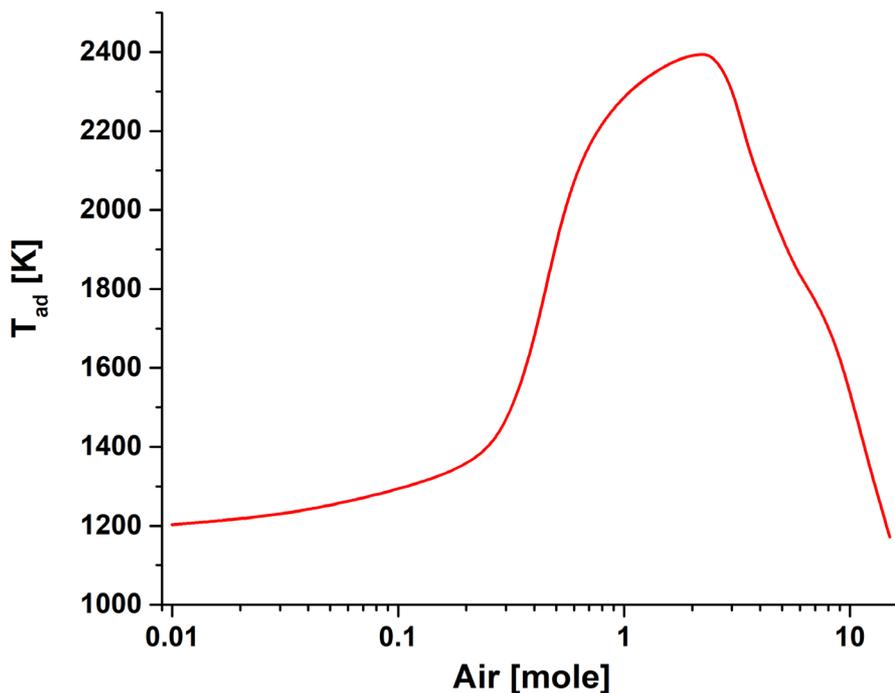


Figure 3. T_{ad} for the primary gaseous combustion products mixed with different air ratios, calculated with the EkviCalc code [12]

In addition, the predicted radiance values at the corresponding temperatures are too low by two orders of magnitude to yield the actual measured radiant intensity. Hence, it has to be concluded that, unlike previous assumptions, the radiative properties of the combustion flame of P in the infrared range cannot be approximated by a graybody type radiator, but must be based on selective emission.

As the investigated formulation contains a hydrocarbon binder, it has to be taken into account that the emission spectra of these combustion flames yield spectra which are dominated by a strong CO₂ band at $\lambda = 4.4 \mu\text{m}$ and only very weak signals in the 6-15 μm range [30].

The band strength of the 15 μm -CO₂ band is less than 1/10 of that of the intensity of the 4.4 μm band [31]. The intensity ratio between the 3-5 range and the 8-14 μm range in these spectra, $\Theta_{v/II}$, is about 10. It is hence obvious that the radiant energy obtained in the 8-14 μm band in the present investigation cannot stem from CO₂.

5 Conclusions and Outlook

- ◆ The combined DTA/TG analysis of the pyrolant shows that the combustion proceeds in the condensed phase with the exothermic reaction of KNO₃ with both Zr and some P, and a concomitant vaporization of the remaining P.
- ◆ The combustion flame of a P(red) rich pyrolant yields intense infrared radiation in both the 3-5 and 8-14 μm bands. The combustion flame of P(red) cannot be described as a graybody radiator, but must be considered as a selective radiator.
- ◆ Current spectroscopic measurements are therefore addressing the nature of the selective radiation from P(red)-based flames.

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