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# On-site Rapid Detection of Trace Non-volatile Inorganic Explosives by Stand-alone Ion Mobility Spectrometry via Acid-enhanced Evaporization

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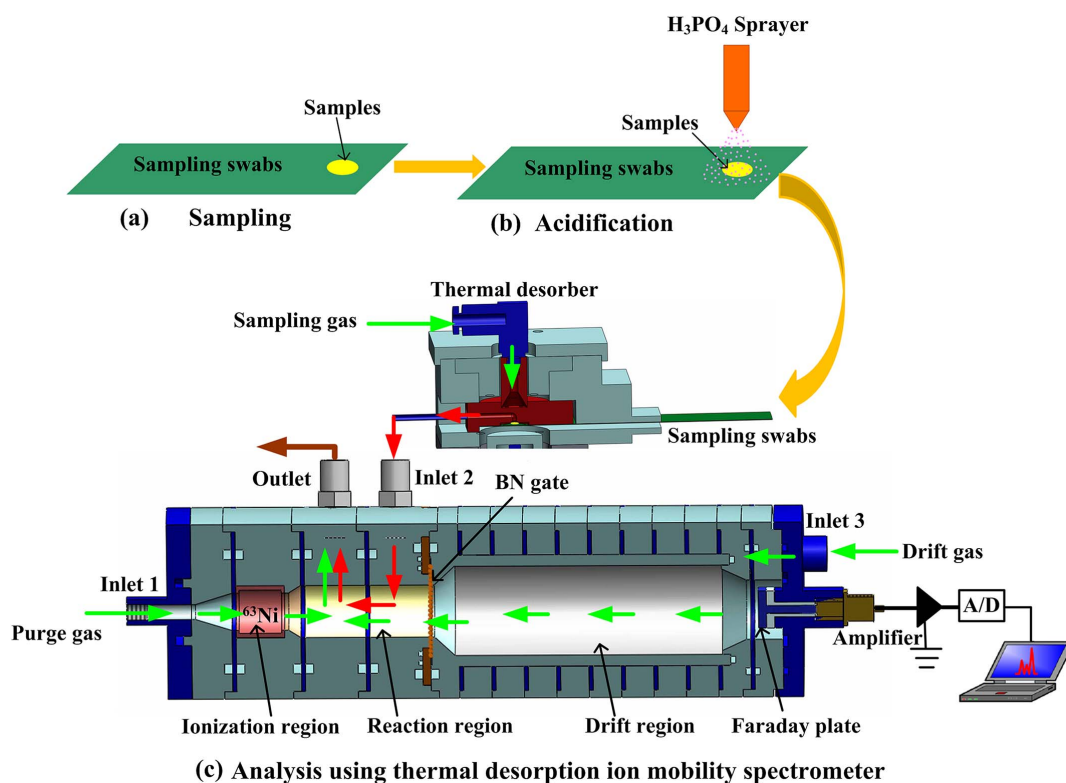
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New techniques for the field detection of inorganic improvised explosive devices (IEDs) are urgently developed. Although ion mobility spectrometry (IMS) has been proved to be the most effective method for screening organic explosives, it still faces a major challenge to detect inorganic explosives owing to their low volatilities. Herein, we proposed a strategy for detecting trace inorganic explosives by thermal desorption ion mobility spectrometry (TD-IMS) with sample-to-sample analysis time less than 5 s based on in-situ acidification on the sampling swabs. The responses for typical oxidizers in inorganic explosives, such as  $\text{KNO}_3$ ,  $\text{KClO}_3$  and  $\text{KClO}_4$  were at least enhanced by a factor of 3000 and their limits of detection were found to be subnanogram. The common organic explosives and their mixtures with inorganic oxidizers were detected, indicating that the acidification process did not affect the detection of organic explosives. Moreover, the typical inorganic explosives such as black powders, firecrackers and match head could be sensitively detected as well. These results demonstrated that this method could be easily employed in the current deployed IMS for on-site sensitive detection of either inorganic explosives or organic ones.

In recent years, rapid and sensitive detection of improvised explosive devices (IEDs) has become the most pressing issues with the increase of the globalization of terrorist acts during the last 2 decades, such as those in Oklahoma, U.S.A. (1995), Bali, Indonesia (2002, 2005), Pune, India (2010), Oslo, Norway (2011), Boston, U.S.A. (2013) and Abuja, Nigeria (2014)<sup>1–3</sup>. On-site screening is an effective way to reduce the risk of explosion, which demands the analytical methods with high requirements in terms of speed, sensitivity, and robustness<sup>4–10</sup>.

The instrumental methods such as X-rays, neutron analysis, nuclear quadrupole resonance, and colorimetric detection are always adopted for the bulk detection of organic and inorganic explosives, while the thermal desorption ion mobility spectrometry (TD-IMS) is proven to be a practical technique for trace detection (from ng to pg) of organic explosives such as 2,4,6-trinitrotoluene (TNT) and cyclo-1,3,5-trimethylene-2,4,6-trinitramine (RDX) *et al*<sup>11–15</sup>. IMS is a gas-phase ion separation and detection technique in a uniform electric field based on the mobility difference of gaseous ions<sup>16–19</sup>. More than 20,000 stand-alone IMS have been deployed at airports and subway stations worldwide for security applications, due to the advantages of fast speed, low cost, analytical flexibility, portability and commercial availability<sup>20–23</sup>. However, it still remains a great challenge for TD-IMS to detect trace inorganic explosives except for ammonium nitrate-fuel oil (ANFO) and sulphur (S) in black powder, owing to their ultra-low vapour pressure even at the typical maximum desorber temperature ( $\leq 280^\circ\text{C}$ )<sup>13,20,24,25</sup>.

Inorganic explosives generally consist of an inorganic oxidizer such as potassium nitrate ( $\text{KNO}_3$ ), potassium chlorate ( $\text{KClO}_3$ ), or potassium perchlorate ( $\text{KClO}_4$ ) and a fuel such as carbon source, sulphur (S), sugar or powdered metals<sup>26,27</sup>. Inorganic explosives are extensively used in terrorist attacks owing to the readily available, low cost and legally purchased components. Ion chromatography (IC)<sup>28,29</sup>, capillary electrophoresis (CE)<sup>30,31</sup>, electrospray ionization mass spectrometry (ESI-MS)<sup>32–34</sup> and electrospray ionization ion mobility spectrometry (ESI-IMS)<sup>35</sup> have been performed to identify their characteristic ions, such as nitrate ( $\text{NO}_3^-$ ), chlorate ( $\text{ClO}_3^-$ ), sulphate ( $\text{SO}_4^{2-}$ ) and perchlorate ( $\text{ClO}_4^-$ ) etc. contained within inorganic explosives<sup>36,37</sup>. However, the field-deployment of these techniques is hampered by the tedious and time-consuming procedures for handling the



**Figure 1** | Schematic of thermal desorption ion mobility spectrometer and the analysis process of inorganic explosives. (a) Sampling: sampling swabs were swiped with samples; (b) Acidification: samples on the swab were sprayed with minimal regime 3%  $\text{H}_3\text{PO}_4$ ; (c) Analysis: the swab was inserted into the thermal desorber and analysed by IMS.

aqueous samples. Additionally, the relatively long analysis time ( $>5$  minutes) of IC and CE, and the relatively large bulk, high expensive cost of mass spectrometer make them not suitable for on-site screening as well.

Obviously, an analytical method which could rapidly and sensitively detect both inorganic and organic explosives is highly demanded. Herein, we firstly reported a simple method to detect inorganic explosives involving  $\text{KNO}_3$ ,  $\text{KClO}_3$  and  $\text{KClO}_4$  using TD-IMS via acid-enhanced evaporation for on-site detection. Samples on the swab were acidized by spraying  $\text{H}_3\text{PO}_4$ , and enhancements of signal for  $\text{KNO}_3$ ,  $\text{KClO}_3$  and  $\text{KClO}_4$  by a factor of over 3000 were achieved, realizing the detection of inorganic explosives at ng even pg level. Moreover, the common nitro-explosives and its mixture with the inorganic oxidizers and the frequently-used inorganic explosives such as black powder, firecracker and match head were sensitively detected. The influence of acidification process on the detection of organic explosives was also investigated.

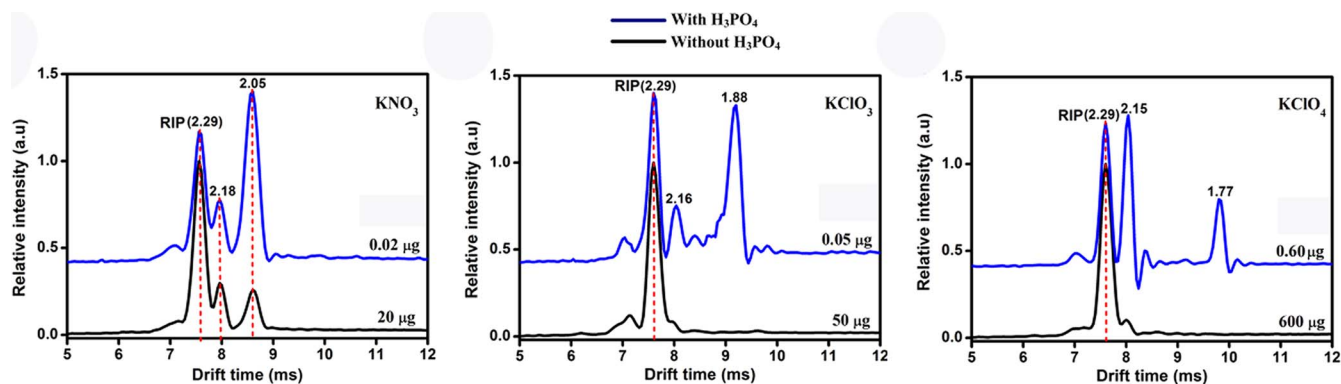
## Results and Discussion

**Design of TD-IMS for analysis.** A swiped-based TD-IMS apparatus operated in negative mode is schematically shown in Fig. 1. The key structure of IMS is similar to those reported in detail previously<sup>25,38</sup> except that an extra inlet named inlet 2 used for the sample inlet was punched in the reaction region at the vicinity of BN ion gate. Thus, only the purge gas flowed through the  $^{63}\text{Ni}$  source, so the pollution and acid corrosion of  $^{63}\text{Ni}$  source could be effectively avoided. In the test, samples were deposited on the swabs firstly (Fig. 1(a)) and then sprayed with minimal regime 3%  $\text{H}_3\text{PO}_4$  (Fig. 1(b)). Afterwards, the swab was inserted into the thermal desorber with temperature of  $180^\circ\text{C}$  for IMS analysis (Fig. 1(c)).

**Enhancement of acidification on the sensitivity.** Ion mobility spectra for typical inorganic oxidizers of  $\text{KNO}_3$ ,  $\text{KClO}_3$  and  $\text{KClO}_4$

obtained without and with 3%  $\text{H}_3\text{PO}_4$  acidification were depicted in Fig. 2, respectively. Clearly, in lack of  $\text{H}_3\text{PO}_4$  acidification on the swab, only two weak product ion peaks with  $K_0$  of 2.18 and  $2.05\text{ cm}^2\text{V}^{-1}\text{s}^{-1}$  were observed for  $20\text{ }\mu\text{g KNO}_3$  and the signal intensity of characteristic peak ( $2.05\text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ ) was only about 83 mV. Whereas, its response for  $0.02\text{ }\mu\text{g KNO}_3$  instantly increased to 280 mV in the existence of  $\text{H}_3\text{PO}_4$ . It was over 3 times higher than that of  $20\text{ }\mu\text{g KNO}_3$  obtained without acidification and the sensitivity were at least enhanced by a factor of 3000. Similarly, no responses for  $\text{KClO}_3$  and  $\text{KClO}_4$  even with quantity of  $50\text{ }\mu\text{g}$  and  $600\text{ }\mu\text{g}$ , respectively were obtained in the absence of  $\text{H}_3\text{PO}_4$ , while two strong ion peaks of  $0.05\text{ }\mu\text{g KClO}_3$  were appeared at  $2.16$  and  $1.88\text{ cm}^2\text{V}^{-1}\text{s}^{-1}$  and peaks with  $K_0$  of  $2.15$  and  $1.77\text{ cm}^2\text{V}^{-1}\text{s}^{-1}$  were observed for  $0.60\text{ }\mu\text{g KClO}_4$  in the assistance of  $\text{H}_3\text{PO}_4$ . And the signal intensities for ion peaks of  $\text{KClO}_3$  ( $1.88\text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ ),  $\text{KClO}_4$  ( $1.77\text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ ) were 184 and 179 mV, respectively. Furthermore, we also adopted other weak acids such as acetic acid ( $\text{CH}_3\text{COOH}$ ) and boric acid ( $\text{H}_3\text{BO}_3$ ) to perform the acidification process. The results demonstrated that  $\text{H}_3\text{PO}_4$  was the most suitable for the analysis due to the perfect sensitivity and its simple background (see Supplementary Fig. S1–2). It is noteworthy that the time taken for sample-to-sample analysis is less than 5 s, and the analysis speed is fast enough for rapid on-site screening.

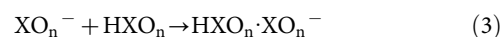
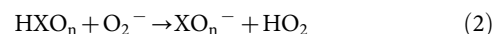
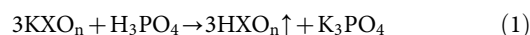
**Mechanism for the acid-enhancement.** The product ions of inorganic oxidizers in  $^{63}\text{Ni}$  source were assigned by an atmospheric pressure negative ionization time-of-flight mass spectrometer (TOF-MS) (see Supplementary Fig. S3). The typical mass spectra were demonstrated in Fig. 3; the reduced ion mobilities ( $K_0$ ), mass-to-charge ratios ( $m/z$ ) and corresponding product ions were summarized in Table 1. Accordingly, two product ions of  $\text{KNO}_3$  at  $m/z$  62 and  $m/z$  125 were assigned to  $\text{NO}_3^-$  and  $\text{HNO}_3\cdot\text{NO}_3^-$ , and



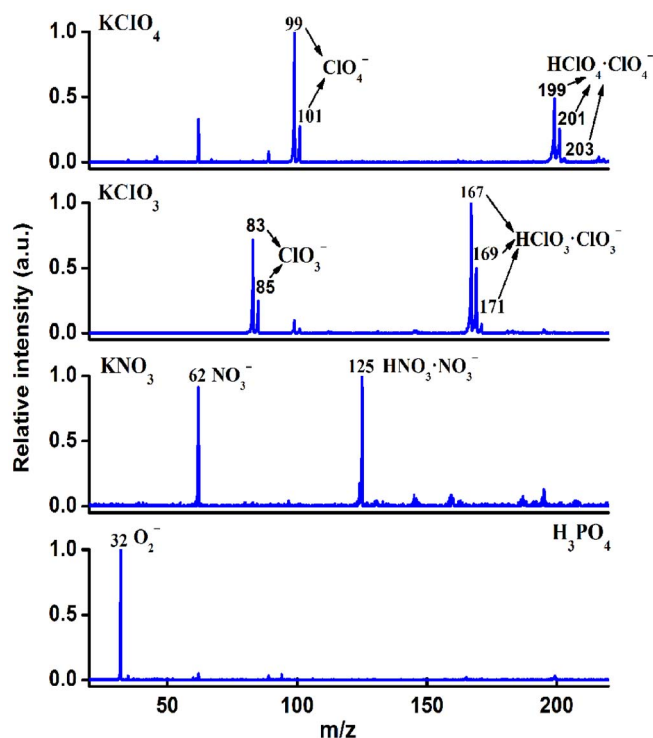
**Figure 2** | Ion mobility spectra of 20  $\mu\text{g}$   $\text{KNO}_3$ , 50  $\mu\text{g}$   $\text{KClO}_3$ , and 600  $\mu\text{g}$   $\text{KClO}_4$  obtained without 3%  $\text{H}_3\text{PO}_4$  and ion mobility spectra of 0.02  $\mu\text{g}$   $\text{KNO}_3$ , 0.05  $\mu\text{g}$   $\text{KClO}_3$ , and 0.6  $\mu\text{g}$   $\text{KClO}_4$  obtained with 3%  $\text{H}_3\text{PO}_4$ .

two ions of  $\text{KClO}_3$  at  $m/z$  83, 85 and  $m/z$  167, 169, 171 were attributed to  $\text{ClO}_3^-$  and  $\text{HClO}_3 \cdot \text{ClO}_3^-$ , while the ions assigned to  $\text{ClO}_4^-$  ( $m/z = 99, 101$ ) and  $\text{HClO}_4 \cdot \text{ClO}_4^-$  ( $m/z = 199, 201, 203$ ) were observed for  $\text{KClO}_4$ . The product ions observed for  $\text{KNO}_3$  were same as those in previous experiments of ammonium nitrate (AN, the main component of ANFO) where  $\text{NO}_3^-$  and complex  $\text{HNO}_3 \cdot \text{NO}_3^-$  were the dominant ions, likely formed by  $\text{HNO}_3$  (the decomposition product of AN)<sup>3</sup>. According to the observed product ions, gaseous inorganic acids, such as  $\text{HNO}_3$ ,  $\text{HClO}_3$  and  $\text{HClO}_4$  were believed to be released in the thermal desorber after acidification through the metathesis reaction between the solid oxidizers and  $\text{H}_3\text{PO}_4$  at high temperature of  $180^\circ\text{C}$ , and then ionized by the reactant ions  $\text{O}_2^-$  in the ion source. Hence, inorganic oxidizers could be indirectly detected by identifying their gaseous inorganic acids in the existence of  $\text{H}_3\text{PO}_4$ . It was further verified by ion mobility spectra of trace  $\text{HNO}_3$  and  $\text{HClO}_4$  (see Supplementary Fig. S4) where the ion peak positions of  $\text{HNO}_3$  and  $\text{HClO}_4$  were same as those of  $\text{KNO}_3$  and  $\text{KClO}_4$  with acidification in

Fig. 2. The mechanism of acid-enhanced detection of inorganic salt oxidizers are summarized as equation (1) to (3) and X presents N or Cl atom.



**Quantitative analysis.** To further investigate the sensitivities of inorganic explosives, we used ion peaks at  $K_0$  of 2.05, 1.88 and 1.77  $\text{cm}^2\text{V}^{-1}\text{s}^{-1}$  for quantitative analysis of  $\text{KNO}_3$ ,  $\text{KClO}_3$  and  $\text{KClO}_4$ . The results were summarized in Table 1; their detectable linear range were 2–60 ng, 10–120 ng and 50–600 ng, respectively and the RSD ( $n = 9$ ) were 17.10%, 16.27%, and 9.99%, respectively. The limits of detection (LOD,  $S/N = 3$ ) for  $\text{KNO}_3$ ,  $\text{KClO}_3$  and  $\text{KClO}_4$  were 0.10 ng, 0.84 ng and 6.30 ng, respectively. These results exhibit the good quantitative capability of the current method for detection of inorganic explosive.



**Figure 3** | TOF-mass spectra of 20 ng  $\text{KNO}_3$ , 50 ng  $\text{KClO}_3$  and 600 ng  $\text{KClO}_4$  obtained with 3%  $\text{H}_3\text{PO}_4$ .

**Detection performances of actual samples.** The acidification process did not affect the detection of organic explosives and typical nitro-explosives such as TNT and RDX could be sensitively detected by current method, and their ion mobility spectra were displayed in Fig. S5 in the Supplementary Information. Clearly, product ion with  $K_0$  of  $1.54 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$  was observed for TNT, and the product ion peaks at  $K_0$  of 1.65, 1.53 and  $1.44 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$  were observed for RDX. Additionally, the mixtures of TNT and inorganic oxidizers could also be sensitively measured as well, and Fig. 4 shows the ion mobility spectra of 5 ng TNT mixed with 20 ng  $\text{KNO}_3$  and 50 ng  $\text{KClO}_3$ , respectively. From the spectra, the characteristic ion peaks of TNT,  $\text{KNO}_3$  and  $\text{KClO}_3$  are clearly observed simultaneously for the mixtures.

Moreover, the oxidizers in typical inorganic explosives were also detected. Figure 5 shows the ion mobility spectra of black powder, firecracker and match head obtained in the existence of  $\text{H}_3\text{PO}_4$ . From the spectra, the characteristic ion peaks of  $\text{KNO}_3$ ,  $\text{KClO}_4$  and  $\text{KClO}_3$  were distinctly observed for black powder, firecracker and match head, respectively. It indicates that inorganic oxidizers in black powder, firecracker and match head are  $\text{KNO}_3$ ,  $\text{KClO}_4$  and  $\text{KClO}_3$ , respectively and other substances in the explosives such as powdered metals and charcoals had little impact on the identification. In particular, only S was detected in the firecracker and black powder without  $\text{H}_3\text{PO}_4$  acidification (see Supplementary Fig. S6), so the detection of both S and inorganic oxidizers all could help identification the materials used in IEDs. Finally, as shown in Supplementary Fig. S7,  $\text{KNO}_3$ ,  $\text{KClO}_3$ ,  $\text{KClO}_4$  in sugar (20 ng  $\text{KNO}_3/\text{sugar}$  (4:1),


**Table 1 |** Reduced ion mobilities ( $K_0$ ), mass-to-charge ratios ( $m/z$ ), product ions and quantitative analysis results obtained with 3%  $H_3PO_4$ 

	$KNO_3$		$KClO_3$		$KClO_4$	
	Peak 1	Peak 2	Peak 1	Peak 2	Peak 1	Peak 2
$K_0$ ( $cm^2V^{-1}s^{-1}$ )	2.18	2.05	2.16	1.88	2.15	1.77
$m/z$	62	125	83, 85	167, 169, 171	99, 101	199, 201, 203
Product ions	$NO_3^-$	$HNO_3 \cdot NO_3^-$	$ClO_3^-$	$HClO_3 \cdot ClO_3^-$	$ClO_4^-$	$HClO_4 \cdot ClO_4^-$
Linear equation	—	$y = 9.83x + 22.90$	—	$y = 5.01x + 2.18$	—	$y = 0.61x + 1.48$
Linear range (ng)	—	2–60	—	10–120	—	50–600
LOD <sup>(a)</sup> (ng, S/N = 3)	—	0.10	—	0.84	—	6.30
RSD <sup>(b)</sup> (% , n = 9)	—	17.10	—	16.27	—	9.99

<sup>(a)</sup>LOD = limit of detection.

<sup>(b)</sup>RSD = relative standard deviation.

50 ng  $KClO_3$ /sugar (4 : 1) and 600 ng  $KClO_4$ /sugar (3 : 2)) could also be sensitively measured, which means that the common fuel in inorganic explosives did not affect the detection.

## Conclusions

We demonstrated a new rapid and sensitive method based on IMS via acid-enhanced detection of oxidizers including  $KNO_3$ ,  $KClO_3$  and  $KClO_4$  to detect inorganic explosives. It owns the advantages of fast speed (about 5 s), low cost, analytical flexibility, portability, commercial availability, and suitable for detecting inorganic explosives and organic ones at the level of ng even pg. Moreover, it is envisioned to be easily deployed to the rapid and sensitive on-site screening of both inorganic explosives and organic ones at airports, subway stations, sports venues and even blast site, being an effective way to screen the substances in the detonation scene and reduce the risk of explosion.

## Methods

**Experiment.** A 500 ng  $\mu L^{-1}$   $KNO_3$ ,  $KClO_3$  and 600 ng  $\mu L^{-1}$   $KClO_4$  standard stock solutions and 3%  $H_3PO_4$  solution were prepared by weighing sample and dissolving in distilled water. The standard solutions and test samples were obtained by gradually diluting the relevant stock solutions with distilled water or other volatile solvents. 20 ng  $KNO_3$ /sugar (4 : 1), 50 ng  $KClO_3$ /sugar (4 : 1), 600 ng  $KClO_4$ /sugar (3 : 2) were made up by mixing  $KNO_3$ ,  $KClO_3$ ,  $KClO_4$  and sugar in corresponding proportion while match head, firecrackers, black powders, 2,4,6-trinitrotoluene (TNT) and cyclo-1,3,5-trimethylene-2,4,6-trinitramine (RDX) were commercial grade.

A known quantity sample solutions were added on or solid samples were rubbed on the swabs firstly and the solvent was evaporated to dryness on a hot plate of 50°C, and

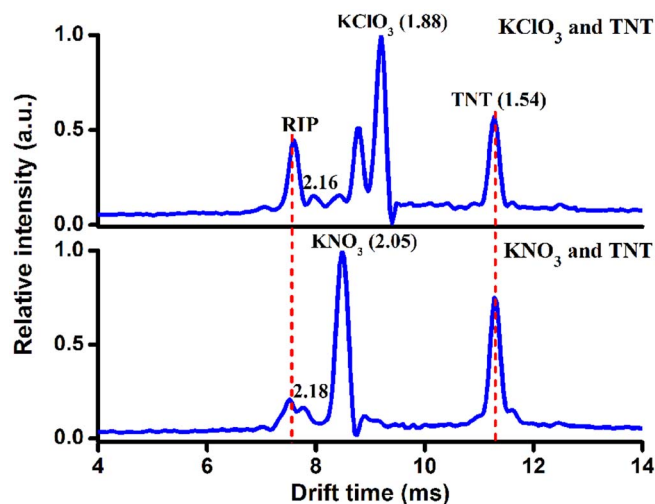
then the swabs with samples were sprayed minimal regime 3%  $H_3PO_4$  (several microlitres). After that the swab was inserted into the thermal desorber for analysis by IMS operated in negative mode. It is noted that enough acid content is essential for the sensitivity.

**Operating process and conditions.** The sample on the swab (2 cm × 8 cm) made of Teflon was heated to 180°C in the thermal desorber and vaporized into a gas flow that passed into the drift tube for analysis. In the  $^{63}Ni$  ionization source, the sample molecules were ionized, and then the product ions were injected into the drift region through the BN gate with an injection pulse width of 300  $\mu s$ . In the drift region, ions were separated according to their mobility with an electric field of 391 V/cm and then detected by the Faraday plate and the ion current was amplified with a gain of  $10^9$  V/A. The drift tube temperature was kept at a constant of 90°C. Air dried by silica gel, activated carbon and 13× molecular sieve traps was divided into drift, carrier and purge gases via flow controllers which were optimized at 800, 400 and 400 mL/min, respectively. The operating parameters were summarized in Table 2.

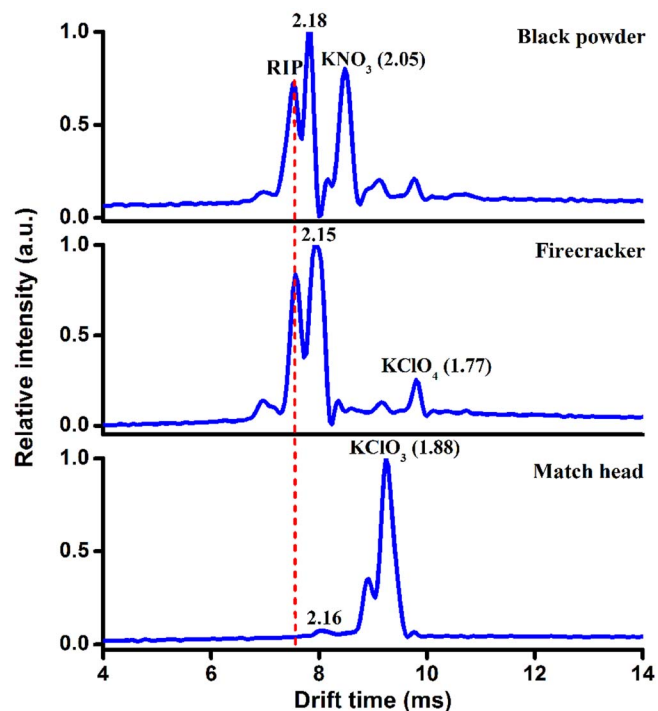
**Calculation.** The reduced ion mobility,  $K_0$  for unknown peaks were calculated with a standard via the following equation,

$$K_{0a} = K_{0s} \times \frac{t_{ds}}{t_{da}} \quad (4)$$

where  $K_{0a}$  is the reduced mobility of the analyte,  $t_{ds}$  and  $t_{da}$  is the drift time of the standard and the analyte, respectively.  $(TNT-H)^-$  was used as the standard substance,



**Figure 4 |** Ion mobility spectra of the mixture of 5 ng TNT and 20 ng  $KNO_3$  and mixture of 5 ng TNT and 50 ng  $KClO_3$  obtained with 3%  $H_3PO_4$ .



**Figure 5 |** Ion mobility spectra of 200 ng match head, 600 ng firecracker and 20 ng black powder obtained with 3%  $H_3PO_4$ .



Table 2 | The IMS operating parameters used in this work

Operating conditions	Setting
Desorber temperature	180°C
Drift tube temperature	90°C
Electric field	391 V cm <sup>-1</sup>
BN gate opening time	300 μs
Carrier gas	Purified Air
Drift gas	Purified Air
Carrier gas flow rate	400 mL min <sup>-1</sup>
Drift gas flow rate	800 mL min <sup>-1</sup>
Purge gas flow rate	400 mL min <sup>-1</sup>

whose  $K_0$  was 1.54 cm<sup>2</sup>V<sup>-1</sup>s<sup>-1</sup> and its drift time was 11.28 ms under the same experimental conditions.

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## Author contributions

L.P. designed the study and wrote the manuscript. L.H. contributed significantly to the measurements on TOF-MS identification. W.W. contributed significantly to the discussion of results and manuscript refinement. Q.Z. contributed to the construction of IMS. H.L. contributed to propose of the project and the refinement of this paper. All the authors contributed to the writing of the manuscript.

## Additional information

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