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Education

A SAFE WAY OF PERFORMING DANGEROUS EXPERIMENTS. III. THE SAFETY BOX. 1. DEMONSTRATING THE REACTIONS OF ALKALI METALS WITH CONCENTRATED ACIDS OF THE TYPE HX(aq) (X=F, Cl, Br, I)

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Earlier publications of the series "A Safe Way of Performing Dangerous Experiments" were devoted to two instances of dangerous experiments and the applied approaches appeared to be safe enough for both the instructor and the audience. Here, we present the results obtained by systematic study of the reactions between Li, Na and K with concentrated water solutions of hydrohalic acids by using a safety box. Video clips of all experiments were prepared as an aid for those that lack experience and/or the necessary materials for safe performance of the demonstrations. Also detailed description and explanation of the observed phenomenon are given.

Keywords: dangerous experiments; safety box; alkali metals; concentrated solutions of hydrohalic acids

БЕЗБЕДЕН НАЧИН ЗА ИЗВЕДУВАЊЕ ОПАСНИ ЕКСПЕРИМЕНТИ. III. БЕЗБЕДНОСНА КУТИЈА. 1. ДЕМОНСТРАЦИИ НА РЕАКЦИИ НА АЛКАЛНИ МЕТАЛИ СО КОНЦЕНТРИРАНИ КИСЕЛИНИ ОД ТИПОТ HX(aq) (X = F, Cl, Br, I)

Претходните публикации од серијата „Безбеден начин за изведување опасни експерименти“ се однесуваа на два вида опасни експерименти, како и на соодветниот пристап кој се покажа достатно безбеден, како за експериментаторот така и за присутните. Сега, пак, се презентирани резултатите добиени со систематско испитување на реакциите на Li, Na и K со концентрирани водни раствори од халогеноводородни киселини, користејќи безбедносна кутија. За сите експерименти, беше подготвен и видео-материјал како своевидна помош за оние кои немаат соодветно експериментално искуство или ги немаат потребните материјали за безбедно изведување на демонстрациите. Исто така е даден детален опис и објаснување на набљудуваните појави.

Клучни зборови: опасни експерименти; безбедносна кутија; алкални метали; концентрирани раствори од халогеноводородни киселини

1. INTRODUCTION

The present paper is a continuation of a series of papers started approximately 15 years ago [1, 2]. For more details, it would be advisable to thoroughly check the contents of the above mentioned publications.

The chemical reactions of the alkali metals with water are well-known [3–13]; however, some videos are not realistic at all [8]. Explosions of pieces of sodium and potassium with water have been demonstrated many times [1, 9–13], but the explanations offered are somewhat questionable [14]. It appears that there is no systematic study of the reactions between alkali metals and concentrated acids. Few videos can be found on the internet, but they lack explanations whatsoever. In order to fill in this gap, as well as to point to a safe way of performing the corresponding demonstrations, this study was performed. In this contribution, we present the results of the reactions of Li, Na, and K with concentrated aqueous solutions of hydrogen halides, namely with HF(aq), HCl(aq), HBr(aq), and HI(aq).

1.1. *Brief review of related works*

The reaction of sodium with water is known to be a very vigorous one (that of potassium is even more vigorous; the potassium and the hydrogen gas are instantaneously ignited even if a small piece of potassium is put in a trough with water). The quantity of heat evolved is large enough to instantaneously melt the metal. Thus, a sphere of molten sodium runs upon the water surface. If the motion of the sodium piece is localized (e.g. placing it over a piece of filter paper floating on the surface of water), then the sodium ignites.

Large pieces of either sodium or potassium can give spectacular results (accounting for the possible side reactions once the metal ignites [9–13]), but **it is dangerous to perform these demonstrations without special precautions**. As noted earlier [1], these reactions would be safe if they could be performed in a hood with the door closed, but under these circumstances, it is not possible to add the metal to the water, unless some device (like the safety spoon [1]) is used.

Quite recently, an original explanation was offered for the explosions of the alkali metals (more precisely, of a liquid alloy of sodium and potassium) in water [14, 15]. The authors mention Coulomb explosion (resulting from the fast transfer of electrons to the water, thus leaving a neat positive charge on the drop of liquid metal) during the explanation. The repulsive forces then simply ‘do the job’. However, in light of the newly proposed demonstrations, it is questionable whether the Coulomb explosion is solely at the origin of this phenomenon [16] the way the demonstration is usually performed. Namely, the authors of the demonstration [14, 15] take special precautions to minimize the surface tension of the molten metals. This is NOT done in a somewhat routine reaction of a piece of potassium/sodium with water; therefore, it is open to discussion whether the explanation works in all possible cases. Experiments of reactions between alkali metals and concentrated acids are also known (video clips are available, together with some brief phenomenological descriptions [17–22]).

The high risk associated with the above experiments stands as a serious obstacle against their use as possible lecture demonstrations. One could imagine that chemical reactions of alkali metals and concentrated acids, in most cases, include even higher risk than the corresponding reactions with water (the reactions with acids are expected to be by far more vigorous). In order to overcome this obstacle, we constructed "The Safety Box" (describes in the Experimental section, 2.2).

1.2. *Facts and expectations*

The solubilities of the reaction products (alkali halides), as well as the strengths of the HX acids in aqueous solutions, differ. The *a priori* expectations would suggest that the stronger the acid and the higher the solubility of the obtained salt in water, the more vigorous the reaction when pieces of the same alkali metal are used with a series of acids. Thus, considering the solubility data of alkali halides (Table 1) and the pK_a values for the four acids (the latter are inversely proportional to the bond strength in the pure hydrogen halides, Table 2), one could *a priori* expect that the reaction with HF should be the least spectacular, and those with HI the most. Further, the reaction of lithium with HF (due to the sparse solubility of the product) is expected to be quite unimpressive.

These expectations were compared with the experimental findings.

Table 1

Solubilities of alkali halides (g/100 g water) at 25 °C [23]

Cation Anion	Li^+	Na^+	K^+
F^-	0.1	4.1	102.0
Cl^-	84.5	36.0	36.5
Br^-	181.0	94.6	67.8
I^-	165.0	184.0	148.0

Table 2

H–X bond strengths and pK_a values of corresponding acids [24]

HX	pK_a	E_{bond}/kJ/mol
HF	3.1	567.7
HCl	– 6.0	431.6
HBr	– 9.0	365.9
HI	– 9.5	298.0

2. EXPERIMENTAL

2.1. *General settings*

Strict precautions were taken for the safety of the experimenter and the handling of the acids and alkali metals. The tweezers and knife used in the experiments were thoroughly dried. All the experiments that were conducted for the first time were performed in a fume hood using the safety box that provided additional safety against possible explosion.

2.2. *The safety box*

To reach the desired level of safety, we constructed a box of acrylic glass and called it a safety box. It can be used in all cases where an explosion hazard exists upon addition of a solid in a reaction system (as in a demonstration of the reaction of alkali metal and concentrated acid). Figure 1 may help in understanding the safety box design.

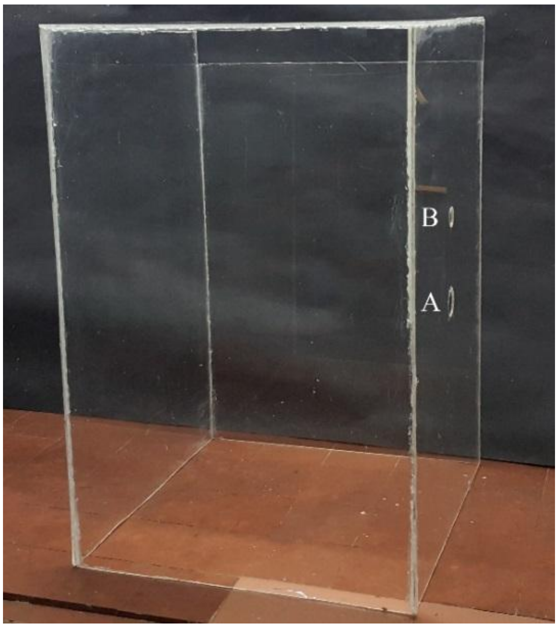


Fig. 1. A safety box made of acrylic glass plates glued together using acrylic glue. The top part of the back plate has an opening in order to avoid pressure buildup in the box during the explosion. Two circular openings (A and B) were placed on the right-hand side plate.

The two small circular openings (A and B) are used for inserting tweezers, regular or safety spoon, or even a regular or safety dropper in the box. This way, the experimenter is fully protected from a possible explosion or sudden splash of the reaction mixture while inserting the alkali metal in the tube or when water is being added. The dimensions and the design of the safety box can vary depending on the needs of the experiment and the setup, the size of the lecture room, the audience, and the purpose. In our case, we used a box with a base that was 40 × 40 cm and height of 60 cm (Fig. 1).

2.3. *Demonstrations: some details*

Most of the mentioned experiments were performed using the safety box. When using sodium or potassium, we took ≈100 mg of the metal, while in the reactions with lithium some 30 mg were used (less because lithium is lighter and has the lowest molar mass, so the quantity is comparable to that of the sodium used).

A test tube with 5 ml of acid was clamped and placed in a jar (the latter can be optionally filled with water to dampen the effect of a possible explosion). All of the above were then placed in the safety box (Fig. 2). The safety box often works well, but if the explosion is very violent (as is the case with nitric or perchloric acid [25]), the test tube and the glass jar can crack.

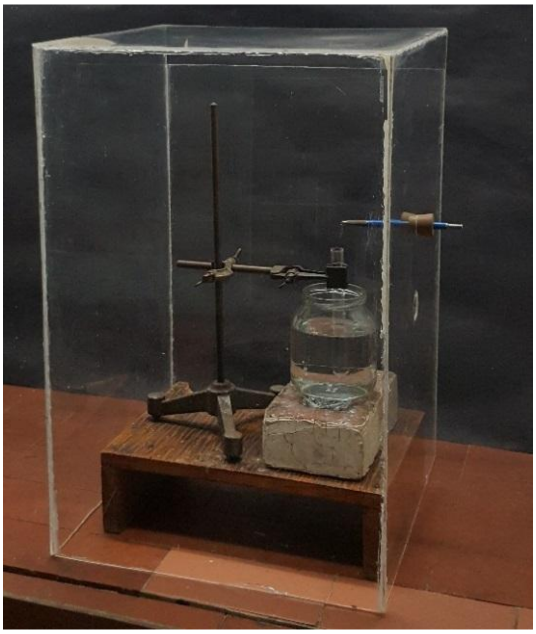
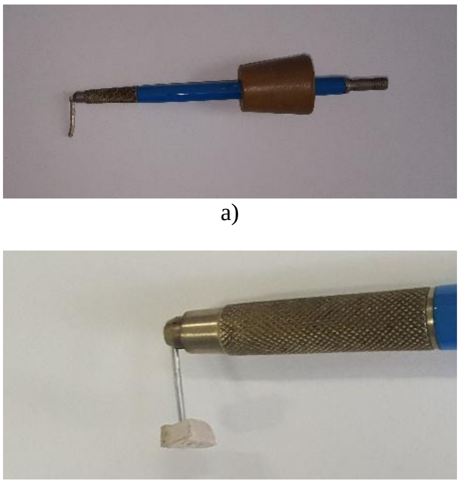


Fig. 2. Complete setup for most of the experiments described in this paper. Using a metal clamp and boss head, the test tube was mounted on a metal stand and immersed in a water-filled jar. The entire apparatus positioned on a wooden stand was placed inside the safety box.

The metal was added using a glass spoon (specifically designed for the safety box and built by a glassblower) from opening A on the box (Fig. 1). Alternatively, a mechanical pencil (Fig. 3a) passing through a rubber stopper could be inserted in opening B. A metal pin (cut from a piece of paper clip) was caught in the pencil's mouth, and the alkali metal was poked on the top of it (Fig. 3b).



a)

b)

Fig. 3. a) Mechanical pencil used for holding the piece of alkali metal and b) the piece of alkali metal pinned on a metal piece cut from a paper clip.

The metal pin was released when needed, thus falling in the test tube with acid and enabling the reaction of the metal with the acid, all while the demonstrator was protected. The presence of the iron from the clip can simply be disregarded.

In cases where experience has shown that the reaction is safe, one could work without using the safety box. This, however, has to be checked experimentally.

When studying the reactions with HF, a plastic test tube was used because glass dissolves (chemically) in hydrofluoric acid according to the following chemical reaction (similar reactions proceed for other silicates):

$$Na_2SiO_3(s) + HF(aq) = 2NaF(aq) + H_2O(l) + SiF_4(g)$$

2.4. *Chemicals*

All chemicals were of reagent grade purity. The acids used in the experiments were concentrated water solutions of the corresponding hydrogen halides with mass fractions in the range of 37–57 %. All were commercially available. The data for the acids given by the manufacturer is shown in Table 3.

Table 3

Assay, density and molar mass data for the aqueous solutions of the hydrogen halides

HX	w/%	ρ/(g cm^{-3})	M_r	Grade
HF	50	1.15 (25 ºC)	20.01	for analysis
HCl	37	1.19 (25 ºC)	36.46	for analysis
HBr	48	1.49 (20 ºC)	80.91	for synthesis
HI	57	1.70 (20 ºC)	127.92	for synthesis

The lithium metal was taken from a freshly opened airtight container filled with argon and used as it was with no additional purification. Sodium and potassium were kept under petroleum. For each reaction, a freshly cut piece was used. The corrosion coating was removed by cutting off the side parts. The petroleum was removed by filter paper, and the freshly cut piece was washed with hexane to remove the petroleum. In Table 4, some data for the alkali metals is given from the manufacturer.

Table 4

Assay, density and molar mass data about the alkali metals given by the manufacturer

Metal	w/%	ρ/ g cm^{-3}	M_r	Grade
Li	≥ 99	0.53 (20 ºC)	6.94	for synthesis
Na	≥ 99	0.97 (20 ºC)	22.99	for analysis
K	98	0.86 (25 ºC)	39.10	for synthesis

3. RESULTS AND DISCUSSION

All reactions discussed in this paper were relatively trivial from the aspect of describing the main reaction that takes place. This reaction can be explained using the following general chemical equation:

$$2M(s) + 2HX(aq) = 2MX(s,aq) + H_2(g)$$

3.1. *Reactions with HF*

As mentioned above, the reaction of lithium with aqueous solution of HF is not an impressive one. First, the product LiF adheres partly to the surface of the metal. The consequence is that the pieces of lithium "dance" on the surface of the acid, lasting a rather long time. The heat of the reaction is not high enough to melt the metal, so besides the generation of hydrogen gas and white-blurring of the liquid (due to the LiF precipitate), there are no other phenomena worth mentioning (Fig. 4a). As seen from Table 2, HF(aq) is a relatively weak acid (its strength is comparable with that of citric acid, pK_1 = 3.15). The corresponding video clip may be found in the Supplemental Material under the name Li_HF.mp4.

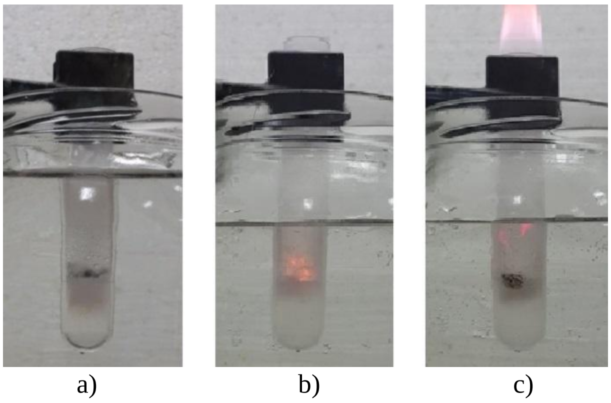

a) b) c)

Fig. 4. Reaction of HF(aq) with a) lithium, b) sodium, and c) potassium. **a)** The piece of lithium metal "dances" on the surface of the HF solution, and a white precipitate of LiF forms in the layer beneath the contact surface where the reaction takes place. **b)** The sodium metal melts and continues to react with the HF solution, occasionally giving sparks, but the hydrogen gas does not ignite. **c)** The evolved hydrogen gas is instantly ignited and the potassium metal melts right away when it touches the surface.

The reaction of HF(aq) with sodium is more vigorous because sodium is more reactive and because NaF is much more soluble in water. Sparks can be seen occasionally in the vicinity of the piece of Na, although the hydrogen gas does not ignite, but the heat released is sufficient to melt the sodium (Fig. 4b). In this case too, white-blurring is evident after a certain period of time (Na_HF.mp4 in the Supplemental Material).

The most vigorous is the reaction with potassium metal. This is, of course, expected since potassium is the most reactive metal compared to the other two (lithium and sodium), and at the same time, the salt that forms is very soluble in water. The metal melts instantly when it touches the surface of HF(aq), and the evolved hydrogen gas immediately ignites (K_HF.mp4 in the Supplemental Material).

3.2. *Reactions with HCl*

The results are, in a way, not very different when one uses concentrated HCl instead of HF. The metallic lithium reacts in a similar manner. It does not melt, and the heat that is released during the reaction is not sufficient enough to ignite the hydrogen that is produced (Fig. 5a). Unlike the reaction with HF, the salt formed in this case (LiCl) is much more soluble (Table 2) compared to LiF (Li_HCl.mp4 in the Supplemental Material).

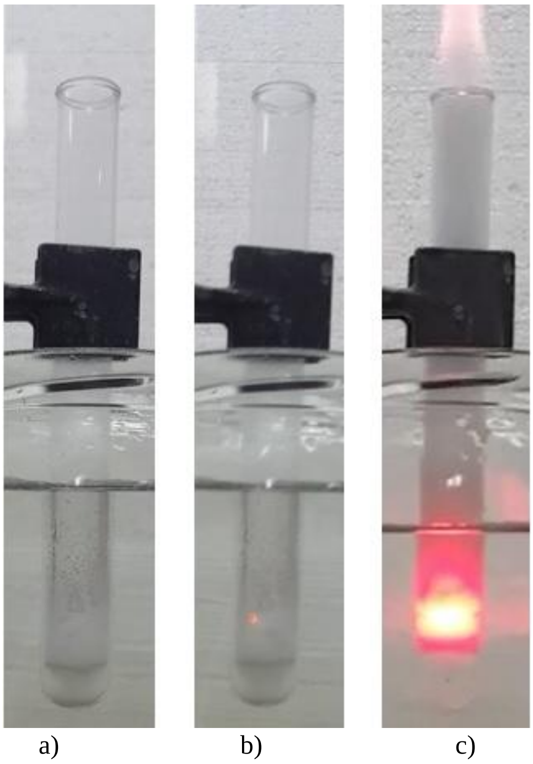

a) b) c)

Fig. 5. Reaction of HCl(aq) with a) lithium, b) sodium, and c) potassium. **a)** The piece of lithium metal "dances" on the surface of the HCl solution. **b)** Sodium metal melts and continues to react with the HCl solution, occasionally giving sparks, but the hydrogen gas does not ignite. **c)** The evolved hydrogen gas is instantly ignited and the potassium metal melts immediately, also igniting.

The reaction of sodium with concentrated HCl is faster compared to the reaction of sodium with concentrated HF. In this case too, a precipitate in the upper layer originating from NaCl is formed during the reaction (Na_HCl.mp4 in the Supplemental Material). The solubility of NaCl is lower compared to the solubility of LiCl, and compared to the solubility of KCl, the same effect can be observed when potassium reacts with a concentrated solution of HCl (Fig. 5b).

The reaction of potassium with concentrated HCl is more vigorous than the reaction of other metals with HCl and also more vigorous when compared to the reaction of potassium with HF. This is expected since potassium is more reactive and, at the same time, HCl is a stronger acid then HF. During the reaction, the potassium metal not only melts, but also ignites spontaneously. At the same time, the produced hydrogen also ignites (K_HCl.mp4 in the Supplemental Material).

The reason why the precipitates NaCl and KCl are formed is due to a combined effect of low solubility (approximately 36 g/100 g H_2O for both salts) and the common ion effect that decreases the solubility of the precipitate. By addition of distilled water, the precipitate dissolves. In order to reduce the common ion effect, the acid was diluted by addition of distilled water, and it was also noted that when the acid was diluted, the reaction became more vigorous, followed by an explosion even in the case of sodium. One of the reasons for this result is that by diluting the acid, the concentration of the chloride ions is reduced and the activity coefficients increase. By lowering the concentration of the chloride ions, the common ion effect is not so pronounced, and the precipitate that forms is readily dissolved in the diluted acid, so that the molten metal comes in contact with fresh acid, unlike in the case of concentrated acid where the precipitate restrains the molten metal to react.

3.3. *Reactions with HBr*

All the reactions of lithium, sodium, and potassium with HBr are much more vigorous compared to the reactions of the same metals with HF or HCl. This is expected due to the increasing strength of the acid compared to the strength of HF and HCl, as well as of the solubilities of the corresponding bromides formed during the reaction (Table 1 and Table 2).

Hydrobromic acid is the first one in the series of hydrohalides that reacts with lithium more violently. In this case, the released energy is sufficient to melt the metal (Fig. 6a). Since the reaction is more vigorous and the lithium is light, the molten metal is often ejected after it melts, and it usually ends up on the test tube wall, not having time to ignite. Before being ejected, small sparks can be noted when the metal reacts with the acid, something that was not observed in the reaction with HF and HCl (Li_HBr.mp4 in the Supplemental Material).

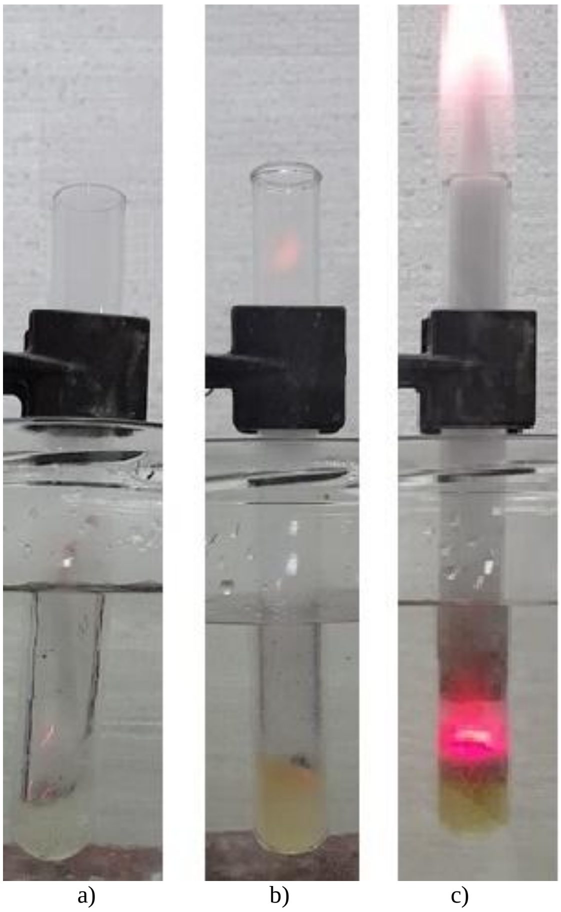

a) b) c)

Fig. 6. Reaction of HBr(aq) with a) lithium, b) sodium, and c) potassium. **a)** The piece of lithium metal melts and continues to react with HBr, sparking before it is ejected. **b)** Sodium metal melts and spontaneously ignites, igniting the produced hydrogen. **c)** The potassium and the evolved hydrogen gas are instantly ignited.

In the case of sodium, the metal not only melts but also ignites, igniting at the same time as the displaced hydrogen gas (Fig. 6b). The same thing happens with potassium, but in this case, the reaction is much more vigorous, and the rate of hydrogen production is higher, making the flame that comes out of the test tube much more intense (Fig. 6c). In all cases, the formed bromides are highly soluble in water, so no precipitate forms.

In the reactions of sodium and potassium with HBr, the faint coloration of the solution during the reaction is interesting and deserves to be explained. The color of the solution turns reddish in the upper layer where the reaction takes place

(Na_HBr.mp4 and K_HBr.mp4 in the Supplemental Material), indicating that elemental bromine is formed. This could be a result of the oxidation of bromide ions into elemental bromine. This does not happen in the case of lithium, while with potassium, it is even more pronounced than with sodium. One possibility is that in the very beginning of the reaction (when air oxygen is present in the test tube), the bromide anions are oxidized to elemental bromine. The brown color soon fades somewhat due to the consumption of bromine in the parallel reaction of the alkali metal with the bromine. Another possibility is that in the first instants of the reaction (when Na or K ignites), the oxidation products contain traces of strong oxidizer (peroxide, i.e. superoxide), which further oxidize bromide anions to elemental bromine.

3.4. *Reactions with HI*

The most vigorous reactions are with hydroiodic acid, as expected. This can be confirmed by the material presented in Figure 7 and the corresponding video clips in the Supplemental Material under the names Li_HI.mp4, Na_HI.mp4, and K_HI.mp4.

The reaction of lithium with HI can be compared to the reaction of lithium with HBr. The reactions are quite similar since the molten metal is ejected in both cases, but the reaction with HI is more vigorous, and the molten metal ignites spontaneously (Fig. 7a1) before being ejected from the reaction mixture. Considering the rate of hydrogen evolution, the reaction with HI is more vigorous, and the same explanation about the ejection of the metal (as for HBr) applies to this reaction as well.

Even sodium reacts with HI explosively. An explosion also happens in the case of the reaction of sodium with HBr, but in the case of HI, it is almost immediate (Fig. 7b2). First, the metal melts, then the released hydrogen ignites (Fig. 7b1), which is followed by an explosion.

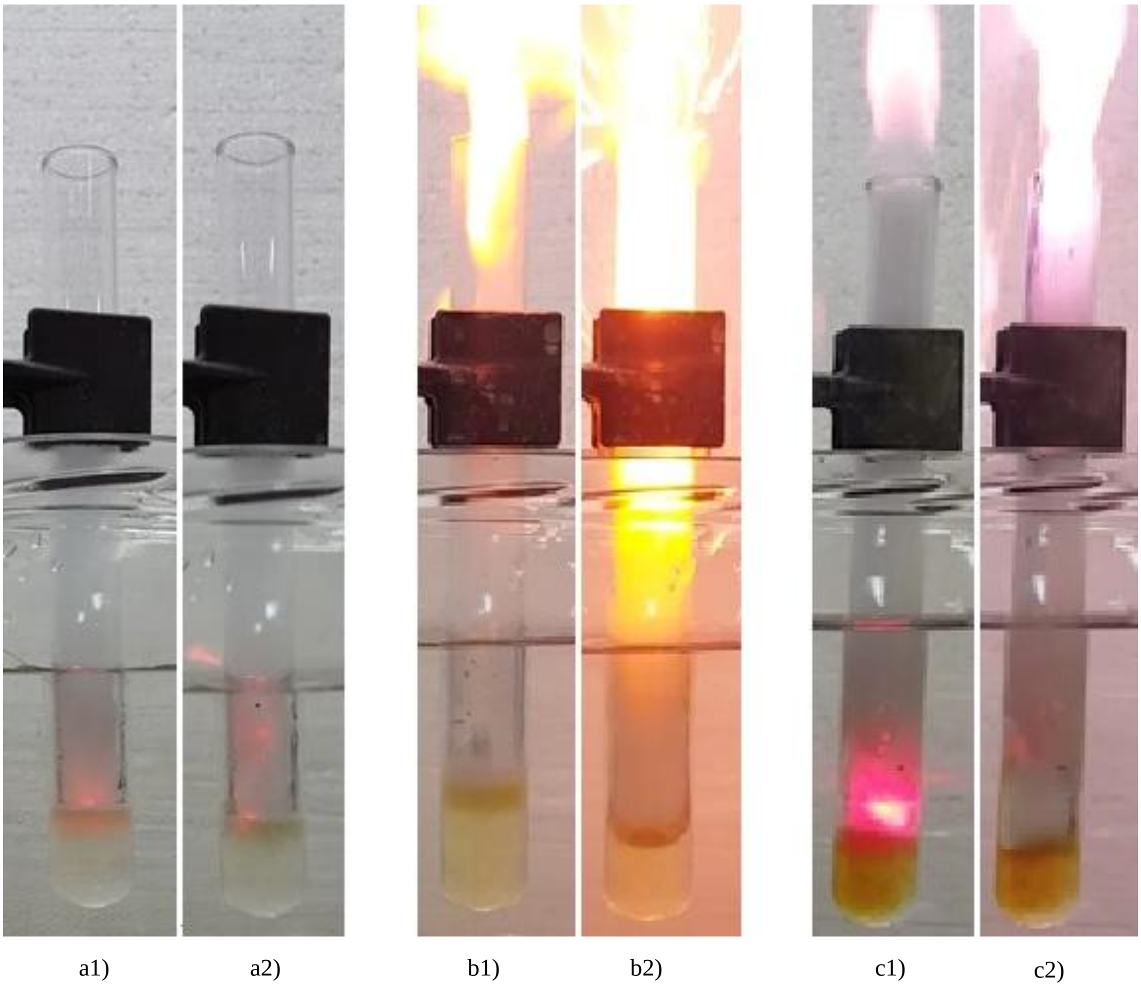

a1) a2) b1) b2) c1) c2)

Fig. 7. Reaction of HI(aq) with a) lithium, b) sodium, and c) potassium. **a)** The piece of lithium metal melts, then spontaneously ignites and is ejected. **b)** Sodium metal melts and spontaneously ignites, igniting the produced hydrogen, causing an explosion. **c)** The evolved hydrogen gas is instantly ignited and the potassium metal melts right away and explodes.

The reaction of potassium with HI is the most vigorous of this class of reactions. Potassium is the most reactive metal with the most pronounced reductive properties (compared to Li and Na), and HI is the strongest acid (compared to HF, HCl, and HBr). So, it is expected that the reaction would be very vigorous compared to all others. The rate of hydrogen production is the highest, and the reaction ends faster than all of the other reactions.

According to Figure **7**, in the reaction with all metals, the HI solution turns brown. The intensity of the brown color increases from Li to K, with the most intense in the case of the reaction of K with HI. Again, as described for the case of the Na and K reaction with HBr, similar reasoning applies to these reactions. In the reaction of lithium with HI, the formation of a brown color can also be noted, but the intensity of the coloration is far less than in the case of the reaction of sodium and potassium with HI. Namely, hydroiodic acid is thermally less stable than HBr, and it can also be oxidized (in the presence of even weak oxidizers) to elemental iodine by the heat released when the piece of lithium reacts.

Taking into account the reaction of sodium and potassium where the released hydrogen ignites, the color of the flame can be determined according to the corresponding flame tests and the presence of metallic ions (Na^+ and K^+, respectively) that are being excited. The color of the flame in the case of sodium is yellow and in the case of potassium is lilac.

4. NOTES

4.1. *Related demonstrations*

These reactions can be compared to the reactions of the alkali metals with water [1, 3–13], concluding that the reactions with concentrated acids are generally much more vigorous (something that would be expected *a priori*).

4.2. *Important information*

The above demonstrations were performed several times, and the experiments always appeared to be safe. However, it is not recommended nor advised that instructors perform the above experiments **unless** they know exactly what they are doing. We are only offering a **safe way** of performing these demonstrations for the instructor who is ready to carry it out, **providing that he/she takes all safety precautions and performs the demonstration exactly as explained above**. In case a reader is willing to perform it (or any other of the mentioned related demonstrations), but is not completely sure whether he/she has prepared everything as required, additional advise, suggestions, or tips can be provided.

4.3. *Safety tips and hazards*

Sodium and potassium are highly reactive metals, and extreme care should be taken when they are used in any experiment! In the presence of water/acids, the metals may spontaneously ignite or even explode. Always wear gloves and a face shield during the preparation of this demonstration. Sodium oxide, sodium peroxide, potassium peroxide, potassium superoxide, sodium hydroxide, and potassium hydroxide (the first four are products of burning sodium/potassium in air; the latter are formed in the reaction of sodium/potassium with water) are caustic, and skin and eye irritants. The reader is advised to check the safety tips from an earlier contribution [1].

5. CONCLUSION

A systematic study of the reactions between Li, Na, and K with concentrated aqueous solutions of hydrogen halide acids was conducted, and a safer way of performing these dangerous experiments was presented. Detailed explanations about the chemistry and the observed phenomena were offered for each reaction.

It was concluded that all metals react with concentrated water solutions of HX, giving the corresponding salts according to the general equation written previously. The rates of the reactions, as well as their velocities, were determined according to the expectations and are in line with the data from Table 2. The main conclusions support the already known reactivity of the metals and the acid strengths. One could then draw the following conclusions:

(1) The reactivity of the alkali metals (Li, Na, K) increases as the molar mass increases, causing the rate and velocity of the reaction to increase as well.

(2) The strength of the acid contributes to the speed of the reaction. By increasing the strength of the acid, the speed increases.

This means that the reaction of potassium with HI is the most vigorous one, as concluded from the experiment, and at the same time, the safest to execute is the reaction of lithium with HF. Also, the effect of precipitate formation should be taken into consideration and, in this case, it goes in "favor" of the reaction of Li with HF, making it even slower.

For those that are still reluctant to perform this kind of demonstration, the available video clip is a safe alternative. The video clip is, at the same time, educational and serves as a warning for students.

Supplemental Material

All video clips and pictures for the performed experiments were made using a Canon EOS 100D camera and are available as a supporting material. Figures 4–7 are snapshots from the video clips and are given under the following names in the collection of videos under the general name M_HX.mp4):

Li_HF.mp4 (reaction of lithium with concentrated hydrofluoric acid)

Na_HF.mp4 (reaction of sodium with concentrated hydrofluoric acid)

K_HF.mp4 (reaction of potassium with concentrated hydrofluoric acid)

K_HBr.mp4 (reaction of potassium with concentrated hydrobromic acid)

Li_HCl.mp4 (reaction of lithium with concentrated hydrochloric acid)

Na_HCl.mp4 (reaction of sodium with concentrated hydrochloric acid)

K_HCl.mp4 (reaction of potassium with concentrated hydrochloric acid)

Li_HBr.mp4 (reaction of lithium with concentrated hydrobromic acid)

Na_HBr.mp4 (reaction of sodium with concentrated hydrobromic acid)

Li_HI.mp4 (reaction of lithium with concentrated hydroiodic acid)

Na_HI.mp4 (reaction of sodium with concentrated hydroiodic acid)

K_HI.mp4 reaction of potassium with concentrated hydroiodic acid)

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