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Lesson Learned from a Fire during Distillation: Choose the Appropriate Condenser

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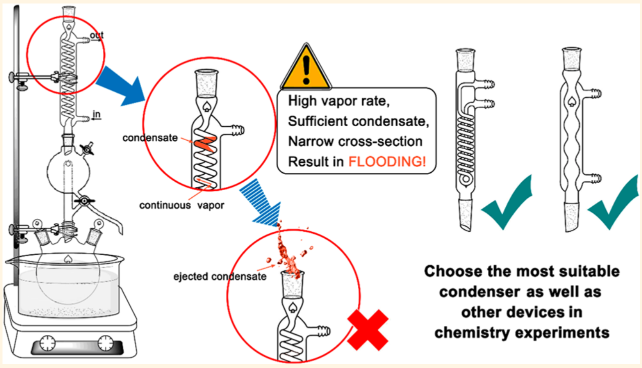
ABSTRACT: A fire broke out during the distillation of carbon disulfide (CS_2). The increasing pressure inside the three-necked flask pushed a stopper open, and the emitted CS_2 subsequently ignited. Fortunately, the fire was put out soon, and no one was injured. In this work, we describe the direct and underlying causes of the incident as well as the subsequent safety conversation about the incident in our massive open online course (MOOC), Safety in the Chemical Laboratory. While the students identified many contributing causes of the incident, none questioned the risk assessment of the experimental setup. Therefore, we provide fundamental information to aid both our students and other researchers in their selection of the most appropriate condenser for their redistillation or reflux conditions.



KEYWORDS: *condenser, distillation, CS_2, fire, chemical safety*

Distillation is a time-honored technique,[1] widely used in the chemistry laboratory to separate mixtures. Additionally, reagent/solvent purification is another distillation application considering that water or other impurities are contained within commercial reagents. For example, commercial carbon disulfide (CS_2) often contains sulfur compounds,[2] so it usually needs to be purified through distillation before use. Nonetheless, several accidents have been reported during distillation and purification, and some have had serious consequences.[3,4]

Here, we report a fire during the distillation of carbon disulfide and a subsequent accident analysis. We also used the accident as part of an online discussion in our massive open online course (MOOC), "Safety in the Chemical Laboratory",[5] to identify the type of information that students need to conduct an effective risk assessment. In response, we underline the importance of a reliable equipment evaluation before experimentation, especially the choice of the condenser used for distillation or reflux.

THE FIRE

To remove the residual sulfur compounds in CS_2, a first-year Chemistry graduate student was redistilling CS_2 in a self-made oil bath setup, which included a transformer, a temperature controller, a heating tube, a stainless-steel basin containing heat transfer oil, and a magnetic stirrer. At 9:50 am, he assembled the device as shown in Figure 1a (without the heating components). In this case, a three-necked flask was used to facilitate the addition of liquid CS_2 during the experiment. After adding commercial CS_2 to the flask, the student turned on the cooling water and the stirrer. The oil bath was then heated and maintained at around 70 °C. At 10:05 am, the solvent still head had collected a sufficient amount of liquid. Subsequently, the student released the liquid from the solvent still head for further use and added about 250 mL of new commercial cold CS_2 to the three-necked flask (1000 mL) directly from the flask's left neck, filling the flask half full. Considering that no abnormality was observed in the device at that time, the student left the lab.

At around 10:30 am, another student in the lab discovered that the distillation device was on fire with one stopper popped (Figure 1b). Fortunately, the fire was put out by a dry powder fire extinguisher, and no secondary damage occurred (Figure 2).

ACCIDENT INVESTIGATION

According to its Safety Data Sheet (SDS), CS_2 is a highly flammable liquid and vapor, and the flashpoint is only −30 °C.[6] Hence, we believe that the fire ignited when a spark from the transformer made contact with CS_2 vapor after the stopper dislodged.

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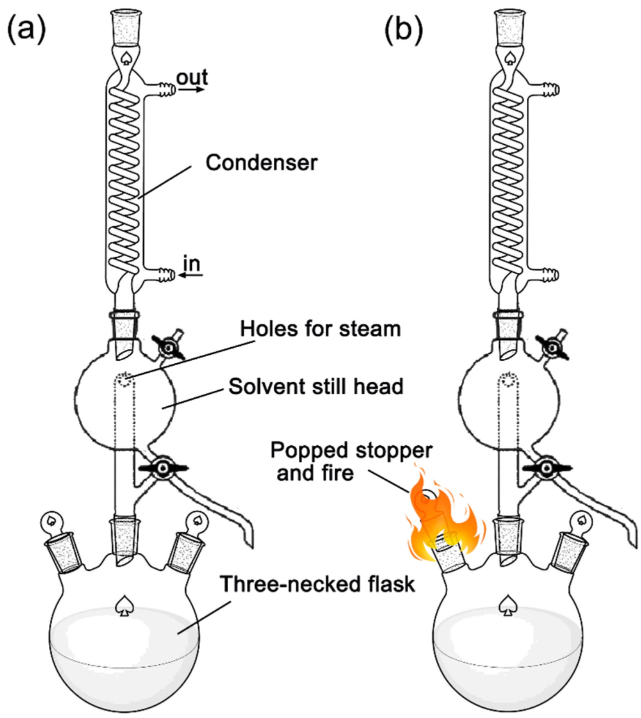

Figure 1. (a) CS_2 redistillation device. (b) Schematic diagram of the fire. The heating device, the stirrer, and the holder are omitted here.

Figure 2. Accident scene after the fire was put out. The note at the front of the fume hood means "Please pull down the fume hood window."

Through investigation, the following factors were also considered:

1. The boiling point of CS_2 is only 46 °C,[6] and the temperature of the oil bath was 70 °C. The relatively high temperature difference would lead to an excessive evaporation rate, which might have increased local pressure in the flask. It is worth noting that the heating temperature normally depends on the reflux ring in the condenser, which should only be from one-third of the way to halfway up the condenser.[7] We cannot confirm the actual situation in this case, but we should state that this temperature setting principle only applies if the correct condenser is selected, and this is discussed later.
2. A Graham condenser was used here. The narrow cross-section of the inside spiral and the relatively high vapor rate can result in flooding; i.e., the condensed liquid fills up part of the inside spiral and is pushed up by the continuous vapor. In doing so, the resulting flood would increase the pressure in the flask until the weakest pressure point is exceeded, which in this case was a stopper.
3. The student was unaware that the fume hood was not turned on during the experiment. It is unknown if the vapor accumulation contributed to this incident or if the initial release of vapor resulted in the fire, but fume hood ventilation can reduce the potential for flammable vapors to accumulate.
4. The transformer used here is not explosion-proof,[8] which could easily lead to a fire in the presence of flammable CS_2 vapor.
5. The student left the laboratory to prepare for another experiment, leaving the highly flammable liquid in a working oil bath unattended. Best practices recommend that distillation setups be continuously monitored.[9]
6. Adding fresh undistilled CS_2 with a heating system on is another inherent risk, which might release the hot flammable vapor. Although the fire was not caused by this, such an operation should be avoided.

■ DISCUSSION

For a further investigation of the origin of this accident and potential mitigations to reduce the impact of a fire, the above reasons will be discussed in reverse order individually or together.

Absence of the Experimenter. Remaining in the lab can enable experimenters to detect an overpressure in a timely manner, to stop heating, and to address a fire immediately. Notably, further analysis would be limited to technical/physical reasons.

Transformer. Devices that might generate sparks should be explosion-proof to avoid fire when flammable vapors might be present.

Fume Hood. In addition to the flammability hazard, CS_2 is neurotoxic,[10] so turning on the fume hood also mitigates a potential toxic exposure risk.

Heating and Condensation. According to previous experiments, with the oil bath at 70 °C, around 200 mL of CS_2 was collected per hour. This rate equals approximately one to two drops per second.

The Graham condenser used here offers much more cooling area than the Liebig or Allihn condenser, which means that the vapor would be cooled and condense well. Notably, though, the condensate (CS_2) may not be able to flow downward against the continuous vapor due to the high evaporation rate and the inside spiral's narrow cross-section. This situation is similar to the flooding in a distillation column at a chemical production facility.[11] This indicates that the mismatch of the heating temperature and the Graham condenser is the main reason for this accident.

To confirm this, we experimented using a simplified equipment setup (Figure 3a), heated by a commercial oil bath. At 55 °C, the reflux of condensate in the Graham condenser was normal. When the oil bath reached 60 °C, the reflux became more active. As shown in Figure 3b and Video S1, flooding started. The red arrow shows one of the places where the condensate filled the inside spiral. Predictably, if the oil bath temperature reaches 70 °C, the flooding will be much more dangerous. Meanwhile, we tested the Allihn condenser, and the flooding did not occur even at 70 °C with the reflux ring between one-third of the way to halfway up the condenser

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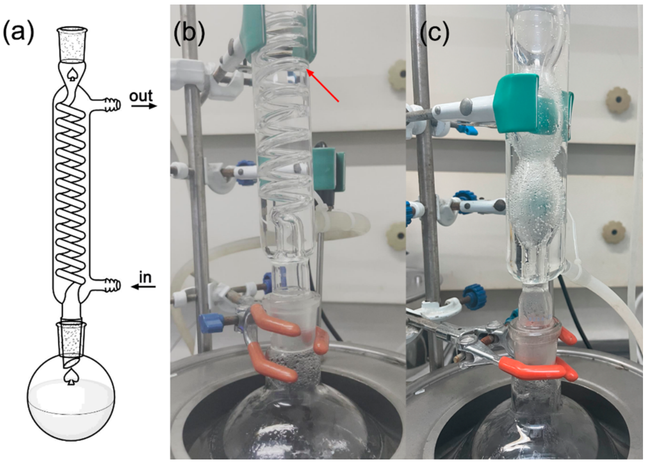

Figure 3. (a) Simplified model equipment for CS_2 redistillation. (b) Flooding in the Graham condenser with the oil bath at 60 °C. (c) Reflux in the Allihn condenser with the oil bath at 70 °C. See details in Videos S1 and S2.

(Figure 3c and Video S2). Hence, we believe that the flood in the condenser resulted in local high pressure in the flask that dislodged the stopper.

Distillation Setup: Plan Your Pressure Relief Points. For distillation or reflux, the constant stream of hot organic vapors would become dangerous if it were in a confined space. Several relevant accidents have been reported: one explosion during distillation was due to a closed valve on the apparatus, which allowed pressure to build up inside,[4] and the other happened when too much urea condensed and blocked the condenser during reaction.[12] The situation in this case is a popped stopper. Tightening the stopper in the three-neck flask or using a standard round-bottom flask might have led to the ejection of condensate (CS_2) from the top of the condenser or some other weak point in the equipment. The ejection of CS_2 could be more dangerous as it might fall into the hot oil bath leading to a fire of conduction oil. Although this implies the risk of using an oil bath (see the guideline in ref 13), this can be minimized by properly designing the distillation setup: for this case, i.e., avoiding the formation of a confined space.

The experimenter did not recognize the risk this experimental setup posed. Their mindset was reinforced by several previous experiments that ran without incident. Specifically, the equipment used was not explosion-proof and the Graham condenser could not accommodate the high evaporation rate of the solvent, thus leading to the overpressure and release of CS_2 vapor.

■ PERSPECTIVE OF STUDENTS

Before determining some strategies to prevent a similar accident, we tried to understand the accident from a student's perspective. For this reason, we used this accident as a discussion topic for our MOOC, Safety in the Chemical Laboratory, where most students were first-year Chemistry graduate students, and others were social learners.[5] In the discussion, the accident was described similarly as in the The Fire section above. Notably, the students were prompted to respond to open-ended questions in writing about what they thought was the reason for the fire and the popped stopper. No detailed options were given to students, and they could see others' answers before participating in the discussion. The discussion lasted for 6 weeks. Several reasons were observed in the students' responses, and Figure 4 shows most of the reasons suggested by students in the fall semester of 2021.

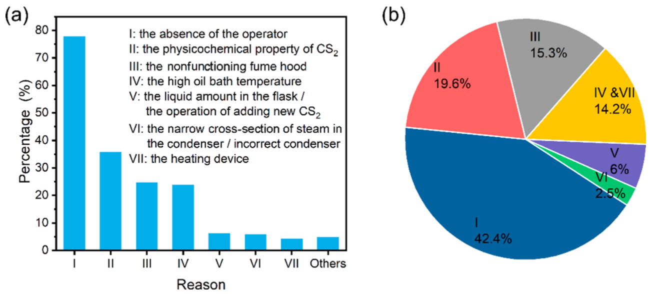

Figure 4. Reasons for this accident as suggested by the students of Safety in the Chemical Laboratory, a massive open online course (MOOC). (a) The reasons that the students mentioned (one students may raise several reasons). (b) The main reason to which the students attributed the fire. Discussions with similar expressions were classified into items I−VII and "Others", as shown in the figure.

The discussion had been browsed 1954 times, and there were 871 valid replies (they were classified into items I−VII). Figure 4a shows the general categories of reasons mentioned and indicates unintelligible discussions in "Other" (i.e., the discussions had nothing to do with CS_2 and the devices), as well as some other rare reasons, such as "the loose stopper".

Figure 4b gives the main reasons raised from the students. Among them, 42.4% of the students thought that the operator did not abide by the laboratory rules, and the absence of the operator was the trigger of the accident (item I). Furthermore, 19.6% attributed the accident to the physicochemical properties of CS_2 (i.e., the low boiling point and flashing point of CS_2 or similar observations), and their analyses stopped here (item II).

On this basis, 15.3% and 14.2% of the students believed that the accident could have been prevented by operating with the fume hood working (item III) or at a lower oil bath temperature (items IV and VII), respectively. Furthermore, only 2.5% mentioned that the narrow cross-section for steam or the incorrect condenser for this experiment led to the accident (item VI).

From the students' discussion, it can be seen that most reasons provided do not reference the best opportunity to identify and mitigate hazards—during the initial risk assessment where potential ignition sources and overpressure should be identified. Hence, the following section will focus on evaluating and selecting distillation setups, especially the condenser choice.

■ CHOOSE THE SUITABLE CONDENSER

For redistillation or reflux, there are many condensers in the lab, such as the Liebig condenser, the Allihn condenser, the Graham condenser, and the Dimroth condenser (return spiral condenser) (Figure 5). The function of a condenser can be divided into two parts, heat transfer (condensation) and mass transfer (reflux). As the heat transfer rate is strongly dependent on the cooling area, an adequate cooling area must be selected.[14] For the four condensers in Figure 5, the Liebig condenser offers the smallest cooling area, followed by the Allihn condenser, the Graham condenser, and the Dimroth condenser, which provides the largest cooling area. As a result, many operators choose the Graham condenser because of its

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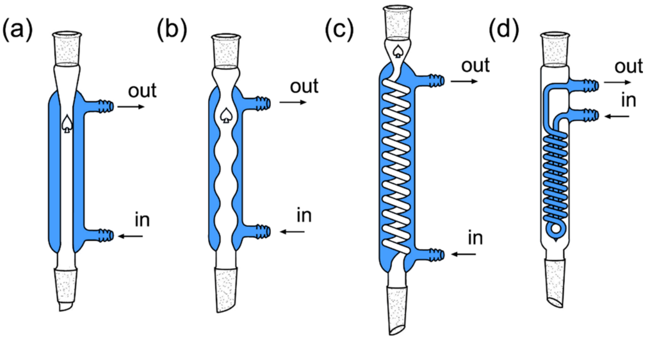

Figure 5. Four common condensers: (a) Liebig condenser, (b) Allihn condenser, (c) Graham condenser, and (d) Dimroth condenser. The blue shading indicates the coolant.

more extensive cooling area. However, mass transfer should also be carefully considered.

For mass transfer in condensers, flooding is a noticeable concern. At high vapor rates, the condensate may not be able to flow down against the rising steam, leading to a liquid ejection from the top or other orifice of the condenser. Notably, such a situation is hazardous. Dalin measured the flood points of different condensers in terms of the wattage input to a kettle.[14] The results showed that the Graham condenser gave the lowest applied wattage, 126 W, when the flood began in the coil, even lower than that of the Liebig condenser.[14] On the contrary, the Dimroth condenser might only flood at the lower joint, which means that the flood point is much higher. The flood points of the four condensers go from the lowest to the highest in the following order: Graham condenser < Liebig condenser < Allihn condenser < Dimroth condenser.[14]

Considering the ability of both heat and mass transfer, we suggest the following condenser choices in distillation or reflux. The Liebig condenser is used only when the vapor rate is relatively low or used diagonally downward for distillation. The Allihn and Dimroth condensers are more favored in reflux or redistillation, and the Dimroth condenser could offer better condensation efficiency. It is worth mentioning that the Allihn condenser should only be used vertically to avoid the condensation being trapped in the bulbs. As for the Graham condenser, it is not recommended to be used for reflux or distillation, especially when the flow directions of the vapor and condensate are opposite. It can only be suitable for the lightest loads, such as the recovery of vapor from permanent gas.

■ CONCLUSION

In summary, we report a fire accident during the redistillation of CS_2. According to our simplified experiment, the mismatch of evaporation rate and Graham condenser resulted in flooding in the condenser, which increased the local pressure and popped the stopper. The emitted CS_2 vapor was ignited by the transformer's electric spark. After discussing with our students in an MOOC, we found that most students (>97%) did not raise the choice of condenser as a potential cause. Hence, we compared the four most common condensers and offer some suggestions on the application range of different condensers. For reflux and redistillation, the Dimroth condenser and the Allihn condenser are more suitable than the Graham condenser. The Graham condenser is recommended to be used only under light loads. Experimenters should evaluate the equipment before the experiment.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.chas.2c00053.

Video S1: Flooding in the Graham condenser at 60 °C (MP4)

Video S2: Reflux in the Allihn condenser at 70 °C (MP4)

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Notes

The authors declare no competing financial interest.

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