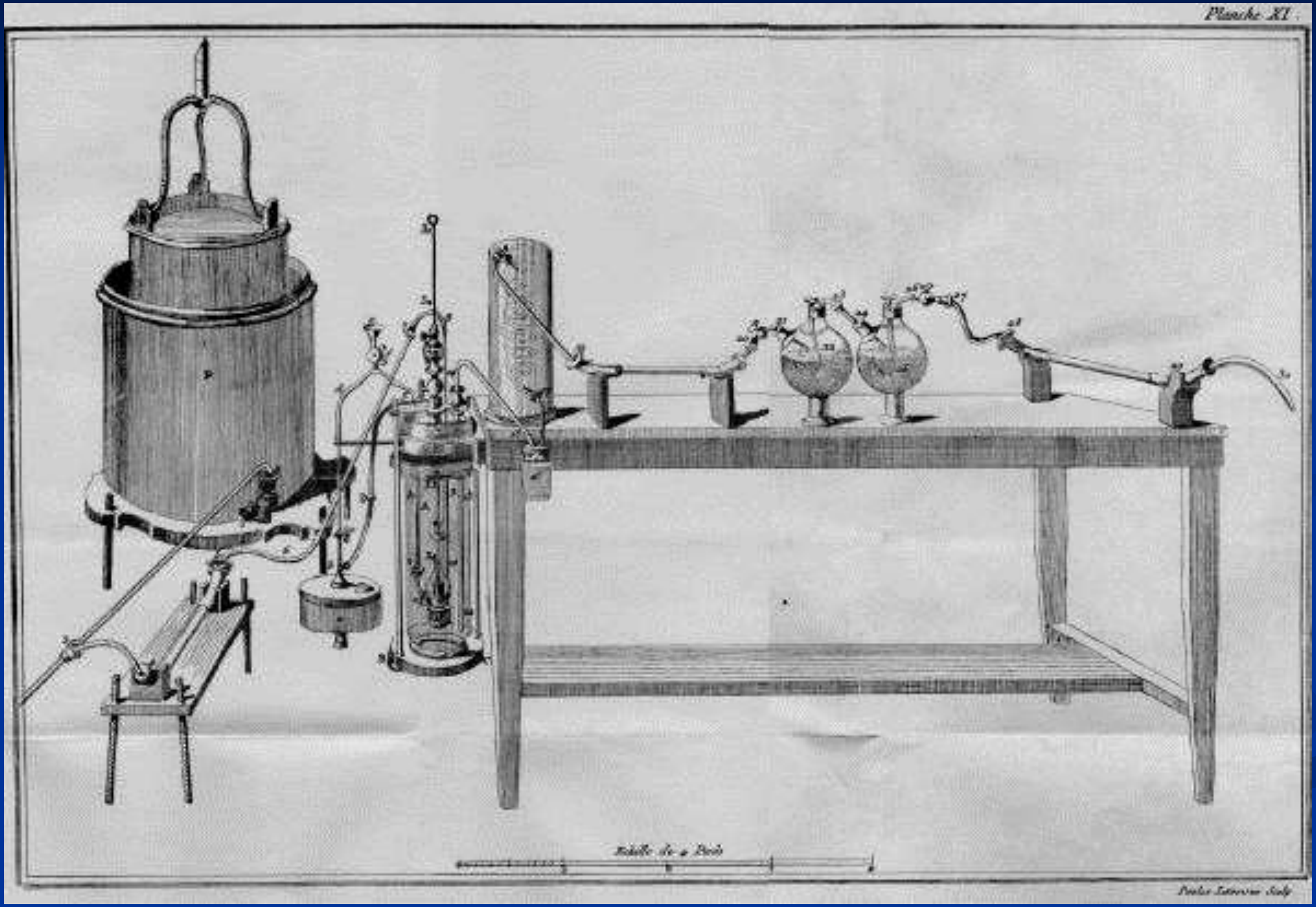
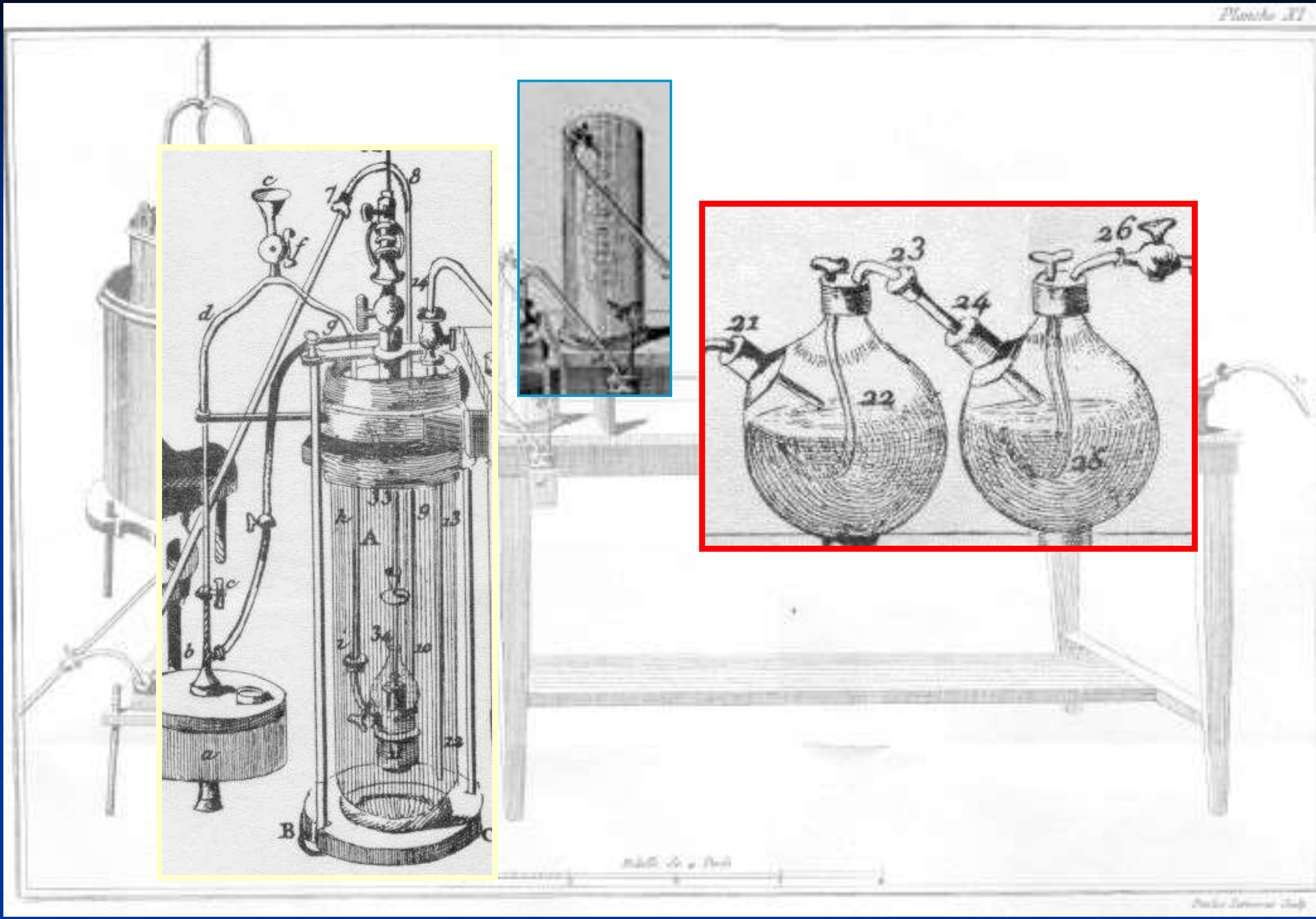
L'analyse élémentaire, du XVIIIè au XXè siècle

Evolution des méthodes d'analyse par combustion

Analyse des huiles par Lavoisier (1785)

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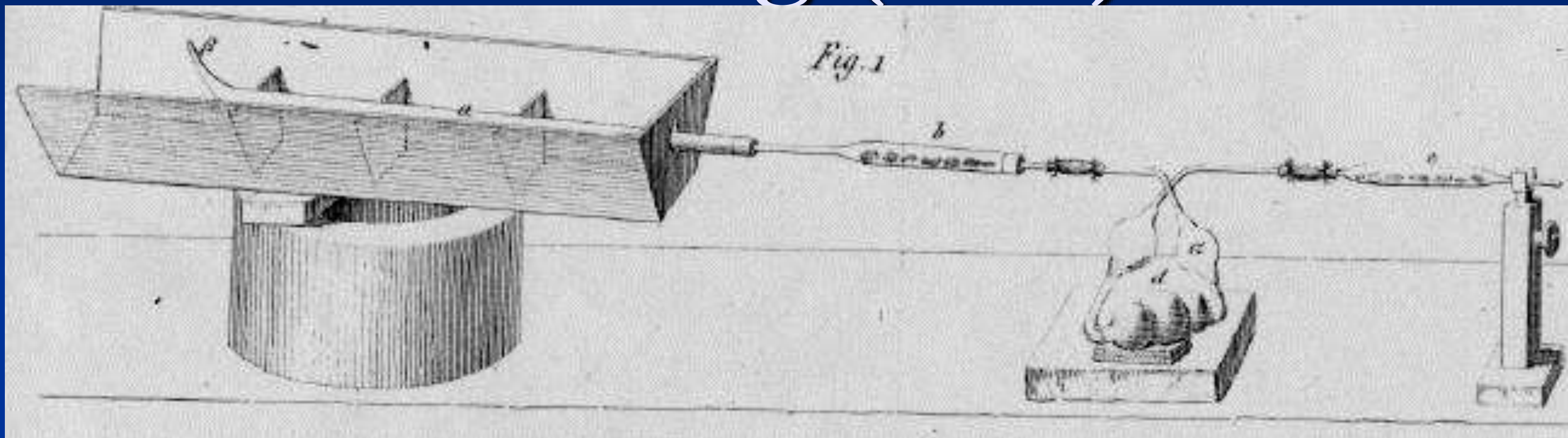
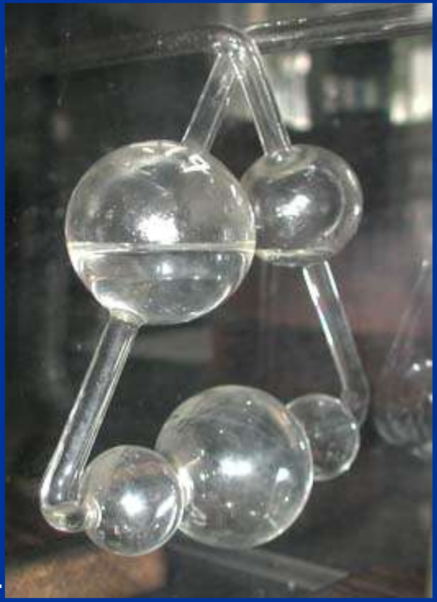
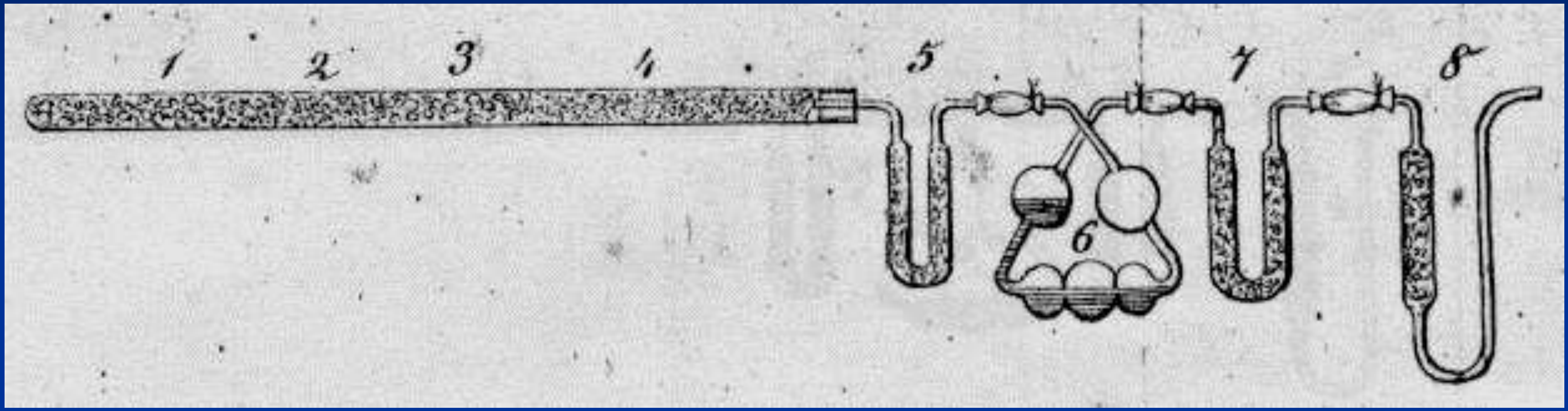
Appareil de combustion de Liebig (1835)

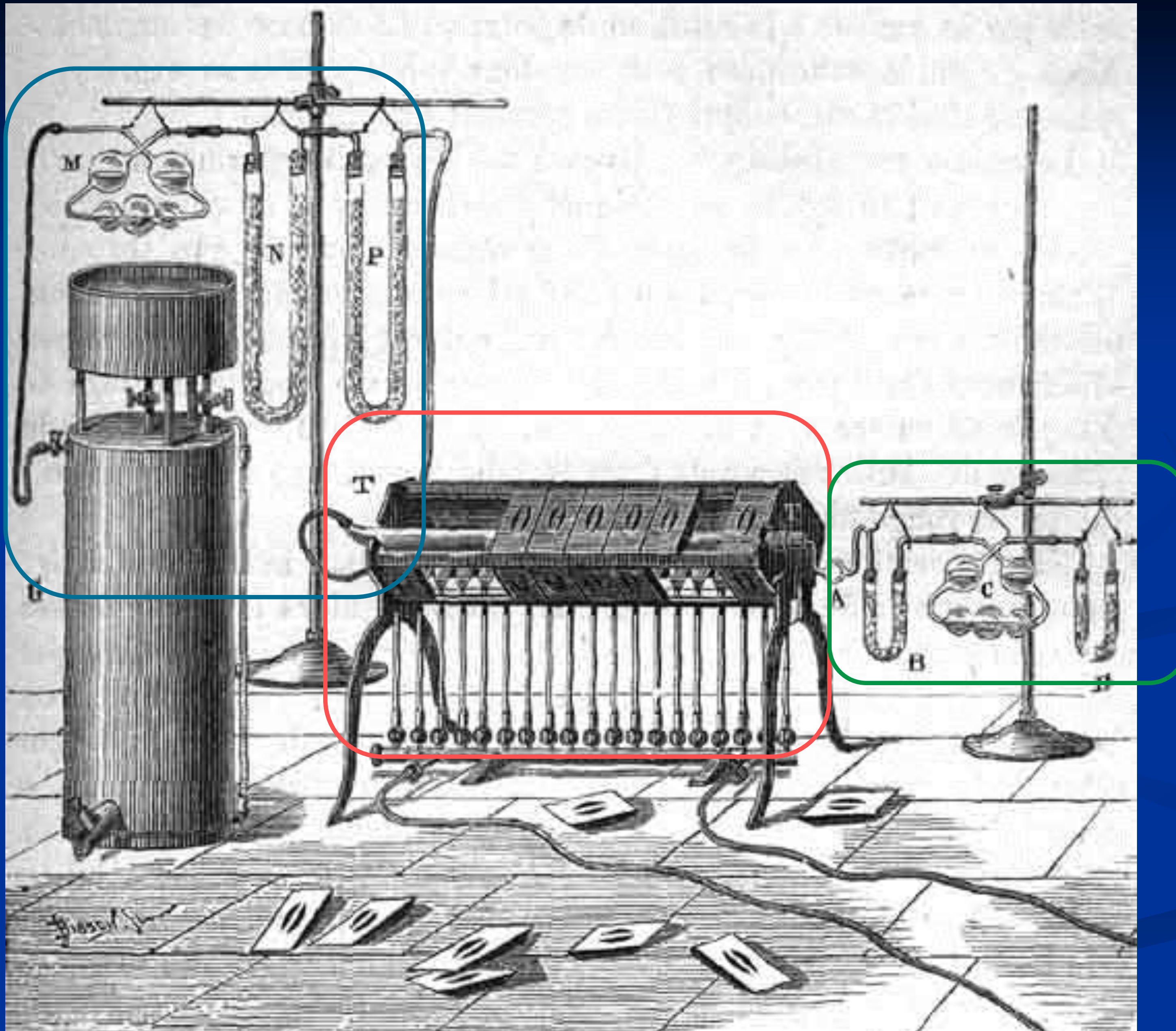




Liebig – Dumas : amélioration 1841



1. CuO and $KClO_3$. When heated this mixture generates O_2 which carries the other gases through the apparatus. This obviates the necessity for breaking the tube at the end of the experiment.
2. CuO
3. Substance mixed with CuO. When heated the CuO converts carbon in the substance to CO_2; hydrogen to H_2O; and nitrogen to N_2. These gases are carried down the train to *5* by the stream of O_2 from *1*.
4. CuO
5. $CaCl_2$ and H_2SO_4. These substances absorb H_2O
6. Liebig's 5-bulb apparatus. As the gas bubbles from bulb to bulb, CO_2 is absorbed by a concentrated solution of caustic potash (KOH)
7. Potassium succinate and dry potash, K_2O. Traps any H_2O and CO_2 that escape from *6*.
8. Aspirator with caustic potash. Keeps atmospheric moisture and CO_2 from entering traps *5-7*.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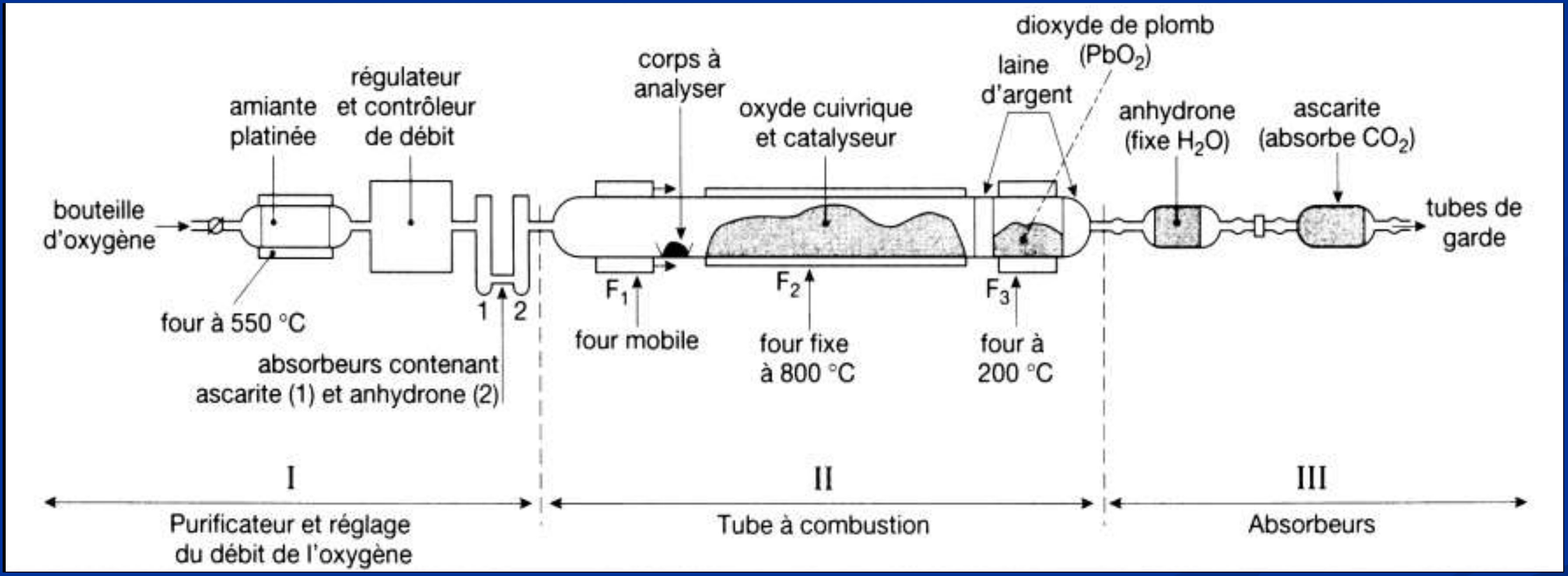
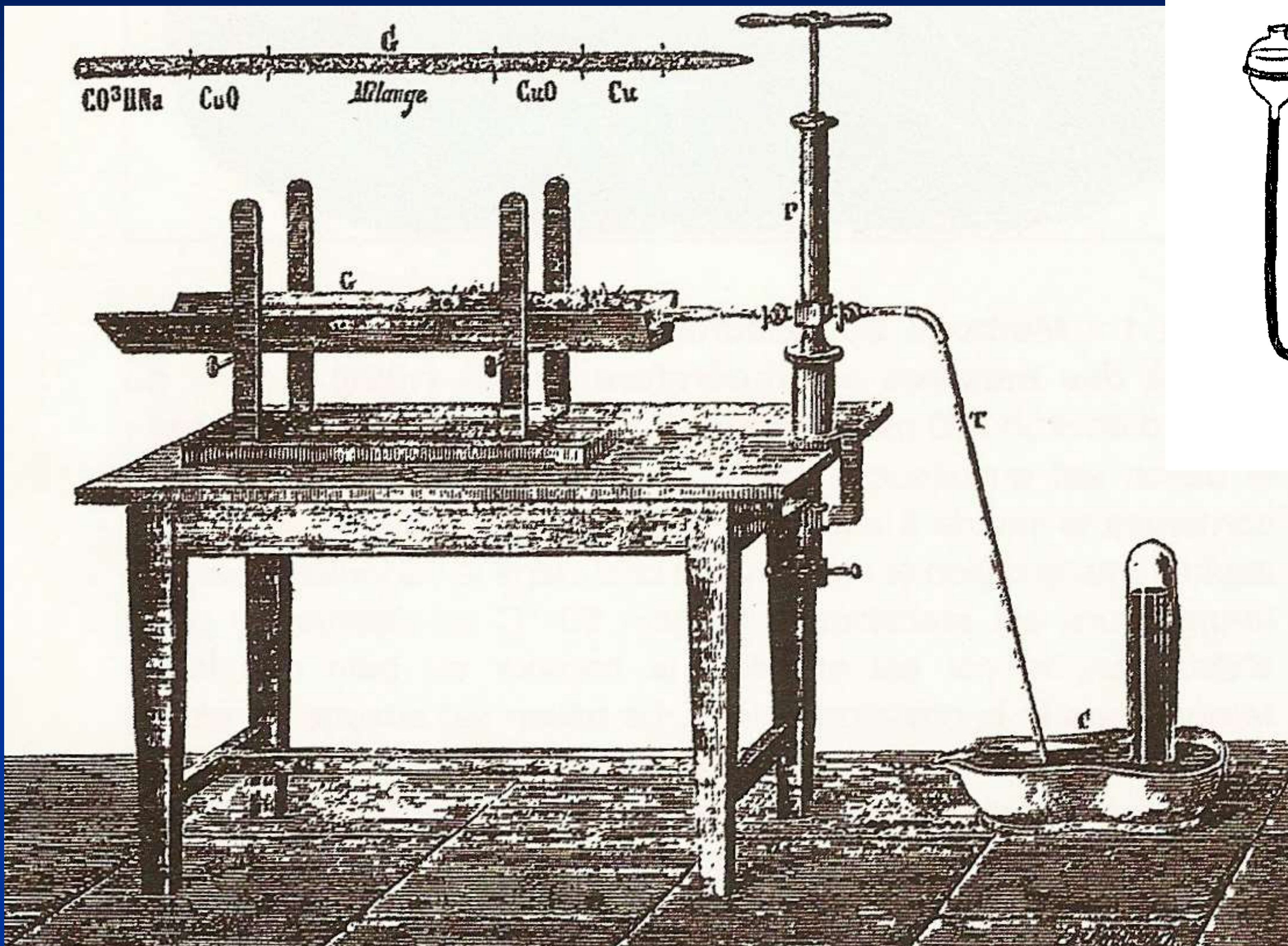
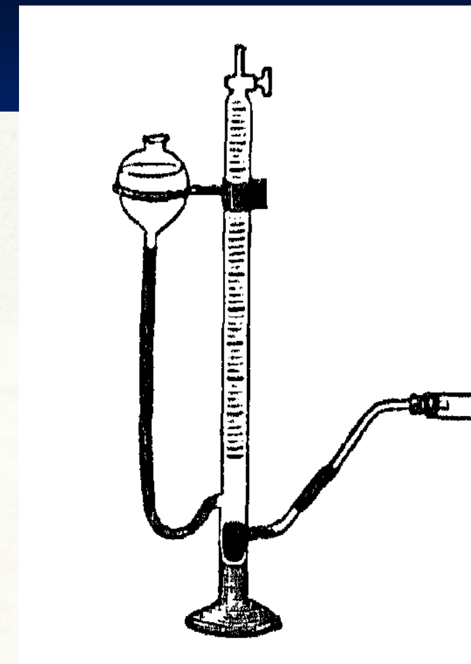
- Principe commun de tous ces appareils



Dumas Azote 1835





Méthode de Kjeldahl (1883)

Minéralisation

Distillation

E. Beauvineau, ENCPB,

Principe de la méthode

- Dégradation de l'échantillon, à l'aide d'un catalyseur et d'acide sulfurique
- Passage en milieu basique, puis distillation de NH_3 pour l'entraîner dans un volume connu d'acide sulfurique (concentration connue)
- Dosage de l'excès d'acide sulfurique par une solution étalonnée de soude

Il nous manque M…

Pour une espèce de formule brute $C_xH_yO_zN_tX_u$:

$$x = \frac{\%C \text{ x } M}{12 \text{ x } 100} \qquad y = \frac{\%H \text{ x } M}{100} \qquad z = \frac{\%O \text{ x } M}{16 \text{ x } 100}$$

$$t = \frac{\%N \text{ x } M}{14 \text{ x } 100} \qquad u = \frac{\%X \text{ x } M}{M_X \text{ x } 100}$$