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THERMOGRAVIMETRY OF COPPER AND COPPER OXIDES (Cu_2O-CuO)

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The thermogravimetry of mixtures of metallic copper and copper oxides was studied. The experiments were performed by heating the samples in air to 700—800° to transform all the components to copper(II) oxide, and continuing the heating in nitrogen to 1050—1100° when the dissociation of copper(II) oxide to copper(I) oxide is complete. The identification of the components and their quantitative determination were carried out by determining the shape, size, and ratio of the segments of the curves obtained during the heating. The method can be used for quantitative analysis of mixtures of copper and/or copper oxides.

In the production of copper powder or copper oxide pigments by different methods as well as in the process of oxidation or corrosion of copper, metallic copper can be accompanied by copper compounds such as: Cu_2O, CuO, $CuCO_3 \cdot Cu(OH)_2$ – malachite, $CuCO_3 \cdot 2\,Cu(OH)_2$ – azurite, or $CuSO_4$.

In order to establish the purity of the product or the mechanism of oxidation, it is important to know exactly which and how many copper compounds the product contains. This paper is concerned with the identification of copper and copper oxides.

Survey of analytical methods

Various methods have been published on the identification and quantitative determination of mixtures of copper with copper compounds. The most important are the following:

1. Chemical analysis, based on the specific solubility of compounds in chemical reagents, the so-called phase-analysis [1–4]. This method gives good results for the separation of sulphides from oxides, but in the presence of CuO or $CuCO_3$ separation of metallic copper from oxides is difficult.

2. Coulometric methods are based on the different behaviour of oxides during electrolytic reduction [5–10]. These methods can be applied only to oxide films having a thickness of up to 2000 Å. It seems that certain difficulties due to the effect of the electrolyte have now been resolved [9, 10].

3. X-ray and electron diffraction methods have so far been applied only to qualitative identification or to crystallographic orientation studies [11–16], but have not been used for obtaining quantitative data.

4. Optical methods: microscopic analysis with reflected, polarized and transmitted light has been used [10, 17–20]. These methods produce good results only for crystalline films, but cannot be used for mixtures of very fine powders.

5. Radioactive tracers: These have mostly been used in kinetic studies, but are not suitable in the quantitative determination of mixtures [21, 22].

6. Thermogravimetric methods: These methods are based on the weight changes during the heating of the sample, and are made in air or in a controlled atmosphere. Such a method has been applied to the study of copper oxidation and also for the analysis of the products [9, 10, 20, 23–35]. For copper oxidation and for the analysis of oxide films Tylecote uses an automatically recording thermobalance, with dry air for oxidation and hydrogen for selective reduction of the two oxides [29]. He considers that copper(II) oxide with its porous structure is first reduced to metallic copper and that only afterwards does the reduction of copper(I) oxide start [32].

7. Differential thermal analysis (DTA) is based on calorific changes (exo or endothermic effects) during the heating of the sample. DTA has, until now, been applied more specifically to define the thermal changes of sulphides, carbonates and oxides, but not for quantitative measurements [33–36].

8. Other methods consist of the measurements of the decrease in pressure of the oxidizing atmosphere in which the specimen is heated, or of the changes in electrical resistance of a wire or thin film due to oxidation [37, 38].

None of the above mentioned methods is entirely satisfactory for the determination of copper compounds. The methods are not sensitive enough if metallic copper is present in a mixture of copper oxides, and particularly if the substances are in form of powders.

Some specific properties of copper and its oxides

a) Metallic copper: The oxidation to Cu_2O starts at room temperature; the process is intensified above 200–250° [26, 27].

b) Copper(I) oxide (Cu_2O), heated in air, starts to oxidize to copper(II) oxide (CuO), visibly at temperatures higher than 250° [26, 27]. The process is intensified at 350°. Above 1025°, copper(I) oxide may dissociate: $2\,Cu_2O = 4\,Cu + O_2$, but the dissociation is very slow until 1250° (P_{O_2} at 1250° is only 0.03 mm Hg; see Table 1) [40, 41, 43].

c) Copper(II) oxide (CuO) may be formed on the surface of metallic copper after long exposure to air, even at room temperature. Above 500–600° CuO decomposes slightly into Cu_2O. This decomposition is intense above 800–900° (P_{O_2} at 1127° is 881.6 mm Hg; see Table 1) [39–41].

The data concerning the temperatures of transformation found in the literature do not always agree [9, 17, 20] and as there are differences of opinion among several authors, we are publishing some data obtained under our own conditions.

Experimental technique

We used a chain-weight balance (AB) (Fig. 1), sensitivity 10^{-4} g, modified by the suspension of a platinum wire (PtW) at the bottom of one scalepan. To this wire a quartz crucible (Cr) of two ml capacity is attached. The crucible is heated in an electric furnace (Fu). The temperature is controlled with a thermocouple Pt–PtRh (TC) fixed near the bottom of the crucible. The whole installation is placed on three shelves (1, 2, 3) covered with asbestos and fixed to a metallic frame. In order to avoid temperature changes in the balance chamber, the distance between the balance and the furnace is one meter. We maintained a constant

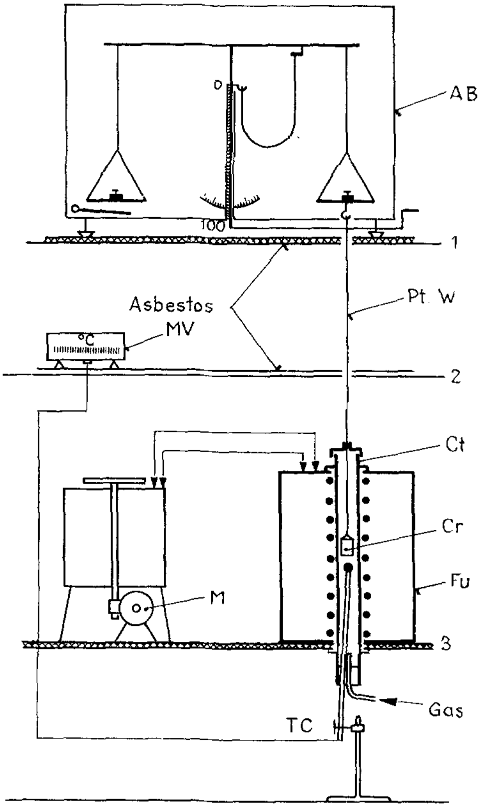

Fig. 1. Arrangement for experiments

temperature with a maximum variation of 2° in the balance case when the furnace was heated up to 1200°. The heating rate of the furnace, 5° per minute, was automatically regulated. The platinum wire passed through the pierced shelves. The quartz tube (Ct) forming the inner part of the furnace was closed at the top by a porcelain plate in which a hole of 3 mm diameter was pierced.

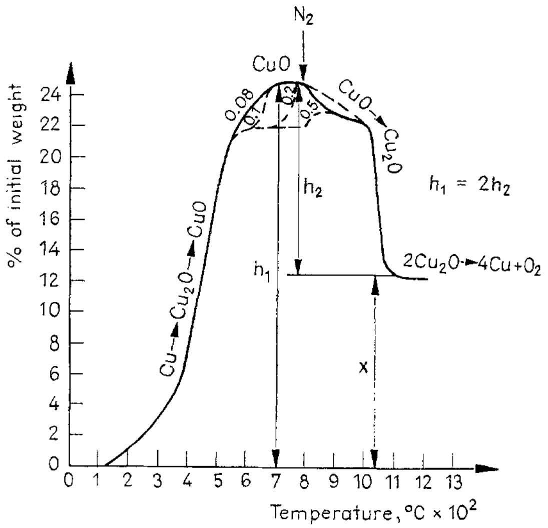

Fig. 2. TG curve of metallic copper

In order to determine the specific characteristics of copper and its oxides during heating, each component was studied separately, and then as binary and ternary mixtures. The first experiments on oxidation and dissociation were made in dried air. Due to the presence of oxygen, the dissociation of copper(II) oxide to copper(I) oxide in air was not complete, therefore purified nitrogen was used in the second stage of the heating (above 700–800°).

To summarize, the method consists of heating the sample, e.g. metallic copper, in dried air at a heating rate of 5° per minute until 700–800°, when a plateau appears on the curve (Fig. 2). With the start of the dissociation of copper(II) oxide we change from air to purified nitrogen, and continue to heat up to 1050–1100° when the dissociation of copper(II) oxide to copper(I) oxide is complete. The slope of the curve changes very sharply at this temperature (1050–1100°) since copper(I) oxide is practically undissociated until 1400–1500° (see Table 1). Changing from air to nitrogen causes a slight variation of weight, but does not affect the results.

Moist substances have to be dried first. In order to avoid oxidation during the elimination of water, heating as well as cooling has to be performed in the furnace of the thermogravimetric balance under nitrogen up to 120 – 180° when a constant weight is obtained with direct reading of lost water.

Table 1

Equilibrium pressures and melting point of copper and copper oxides

a) Equilibrium pressure of the reaction [39] $4CuO = 2Cu_2O + O_2$		b) Equilibrium pressure of the reaction [4] $2Cu_2O = 4Cu + O_2$		c) Melting point, °C
t °C	P, mm Hg	t °C	P, mm Hg	
527	0.0003	1064	0.00014	Metallic copper 1083—1084 [36, 40, 42]
627	0.0	1150	0.0014	Copper(I) oxide 1230—1242 [39, 40, 42]
727	0.11	1250	0.03	Copper(II) oxide 1336—1720 [40]
827	2.14	1350	0.23	
927	21.51			
1027	161.88			
1127	881.60			

Notations:

h_1 weight increases during the oxidation
h_2 weight decreases during the dissociation
x oxygen required by the metallic copper to produce Cu_2O; $2x$ to produce $CuO(Cu_2O_2)$
y oxygen required by initial copper(I) oxide to produce CuO
z oxygen eliminated by dissociation of the initial copper(II) oxide to copper(I) oxide, Cu_2O

If initially we have only metallic copper and copper oxides:

$G_0 = G_{Cu} + G_{Cu_2O} + G_{CuO}$
G_0 = the initial weight of the sample
G_{Cu} = the initial weight of the metallic copper
G_{Cu_2O} = the initial weight of the copper(I) oxide, Cu_2O
G_{CuO} = the initial weight of the copper(II) oxide, CuO

From stoichiometric relations:

$G_{Cu} = 7.94x \simeq 8x$
$G_{Cu_2O} = 8.94y \simeq 9y$
$G_{CuO} = 9.94z \simeq 10z$

Results

I. *Metallic copper (Fig. 2)*

The following materials were used:

(a) Electrolytic copper powder, 99.98 % Cu.

(b) Copper cement sheets and powder of 0.001 – 0.5 mm thick, 99.80 – 99.90 % Cu (impurities: Fe, Fe_2O_3, C, Si less than 0.2 %).

The copper is oxidized up to 600–700°, followed by a very slow decomposition until 900°. At 900–950° the decomposition becomes rapid, and stops at about 1050–1150°. At this temperature a sudden change in slope is observed, which shows the end of the dissociation of CuO and the beginning of the Cu_2O dissociation. From Fig. 2 we can conclude that copper, less than 0.1 mm thick, oxidizes more easily to Cu_2O and CuO. Copper more than 0.1 mm thick has a threshold which becomes more prominent with the increase of the particle size. This threshold is due to oxidation starting at the surface and forming a layer (film) of Cu_2O; the oxidation advances towards the centre. The mechanism of film-growth in the presence of metallic copper is still not precisely established [10, 45–47]. The diffusion of ions from the metal to the oxide film through the oxide-metal interface and through lattice defects, forms the basis of most theories [29–31, 45–49]. Only when the outside is saturated with CuO (oxygen), does the oxidation advance into the depth of the particle, turning the copper completely into copper(II) oxide. If the copper is thick, it is possible that part of the metal does not become completely oxidized, some of it remaining as Cu_2O or even metallic copper. If the oxidation is effected above 800°, the process is superposed on the dissociation of CuO, so that the experiment is no longer conclusive. This fact can be clearly observed from the oxidation curves, so that in all the experiments the size of the particles or of the foils was limited to less than 0.1 mm. Here, complete oxidation occurs up to 700–750°.

It follows that for metallic copper we have (Fig. 2):

$$\mathrm{Cu} \xrightarrow{x} \mathrm{Cu_2O} \xrightarrow{x} \mathrm{CuO} \xrightarrow{x} \mathrm{Cu_2O}$$

$$h_1 = 2x; \quad h_2 = x; \quad h_2 = h_1/2$$

II. *Copper(I) oxide, Cu_2O (Fig. 3)*

$$\mathrm{Cu_2O} \xrightarrow{y} \mathrm{CuO} \xrightarrow{y} \mathrm{Cu_2O}$$

$$h_1 = y; \quad h_2 = y; \quad h_2 = h_1$$

Cuprous oxide was prepared by heating pure copper(II) oxide in purified nitrogen to 1200°, and cooling to room temperature. The oxidation of copper(I) oxide starts very slowly above 250° and more intensely above 300–350°, as reported by others [29].

III. *Copper(II) oxide, CuO (Fig. 4)*

$$\mathrm{CuO} \xrightarrow{z} \mathrm{Cu_2O}$$

$h_2 = z$; $h_1 = 0$ Only dissociation occurs.

Above 700–800° the copper(II)oxide is dissociated to copper(I) oxide. The dissociation is completed at 1050–1100° in nitrogen.

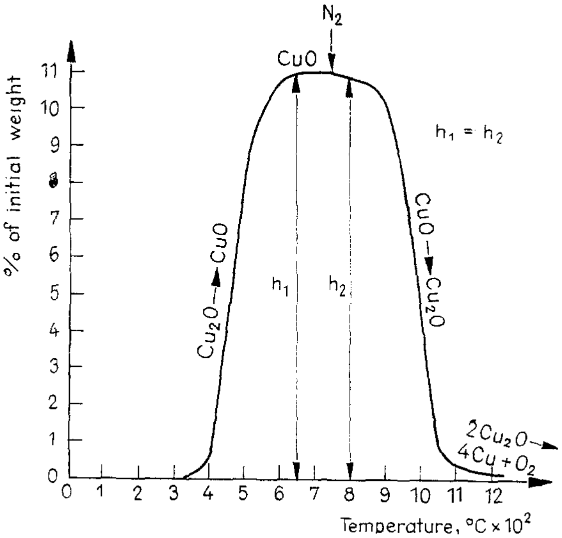

Fig. 3. TG curve of copper(I) oxide

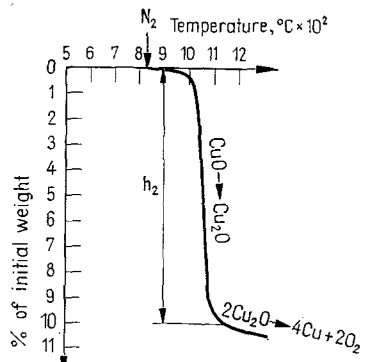

Fig. 4. TG curve of copper(II) oxide

The same technique was used with binary mixtures, namely, heating the mixtures in dried air up to 700–800° and continuing the heating in dried nitrogen up to 1050–1150°.

IV. *Metallic copper and copper(I) oxide, Cu – Cu_2O (Fig. 5)*

(a) $Cu \xrightarrow{x} Cu_2O \xrightarrow{x} CuO \xrightarrow{x} Cu_2O$

(b) $Cu_2O \xrightarrow{y} CuO \xrightarrow{y} Cu_2O$

$h_1 = 2x + y;\ h_2 = x + y;\ h_1 - h_2 = x$

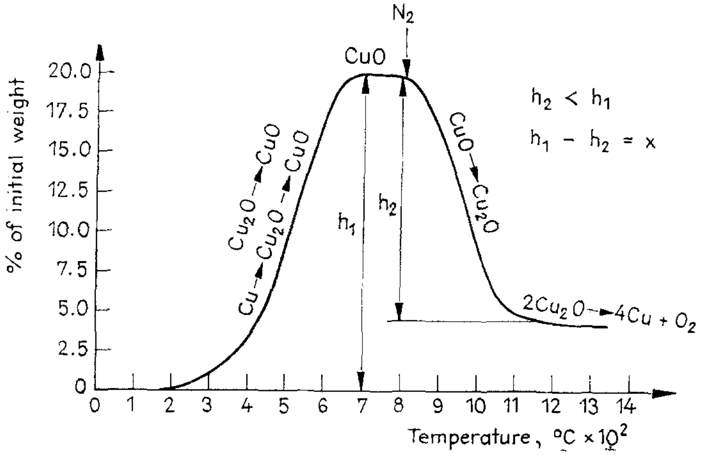

Fig. 5. TG curve of metallic copper and copper(I) oxide

V. *Metallic copper and copper(II) oxide, Cu – CuO (Fig. 6)*

(a) $Cu \xrightarrow{x} Cu_2O \xrightarrow{x} CuO \xrightarrow{x} Cu_2O$

(b) $CuO \xrightarrow{z} Cu_2O$

$h_1 = 2x;\ h_2 = x + z;\ h_1 - h_2 = x - z$

Depending on x and y values, i.e., the quantities of metallic copper and copper(II) oxide, the heating curve may present three aspects:

1. $h_2 < h_1;\ z < x$
2. $h_2 = h_1;\ z = x$
3. $h_2 > h_1;\ z > x$

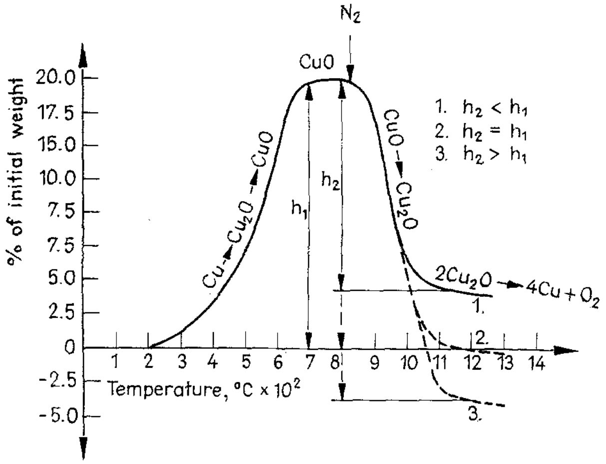

Fig. 6. TG curve of metallic copper and copper(II) oxide

VI. *Copper(I) oxide and copper(II) oxide, Cu_2O-CuO (Fig. 7)*

(a) $Cu_2O \xrightarrow{y} CuO \xrightarrow{y} Cu_2O$

(b) $CuO \xrightarrow{z} Cu_2O$

$$h_1 = y;\quad h_2 = y + z;\quad h_2 > h_1;\quad h_2 - h_1 = z$$

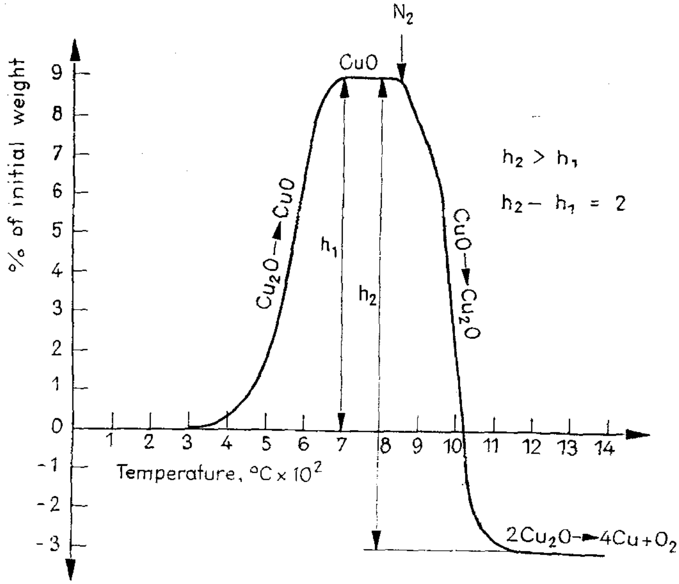

Fig. 7. TG curve of copper(I) oxide and copper(II) oxide

VII. *Ternary mixtures. Mixtures of copper and copper oxides: Cu – Cu$_2$O – CuO (Fig. 8)*

(a) $Cu \xrightarrow{x} Cu_2O \xrightarrow{x} CuO \xrightarrow{x} Cu_2O$

(b) $Cu_2O \xrightarrow{y} CuO \xrightarrow{y} Cu_2O$

(c) $CuO \xrightarrow{z} Cu_2O$

$h_1 = 2x + y; \quad h_2 = x + y + z$

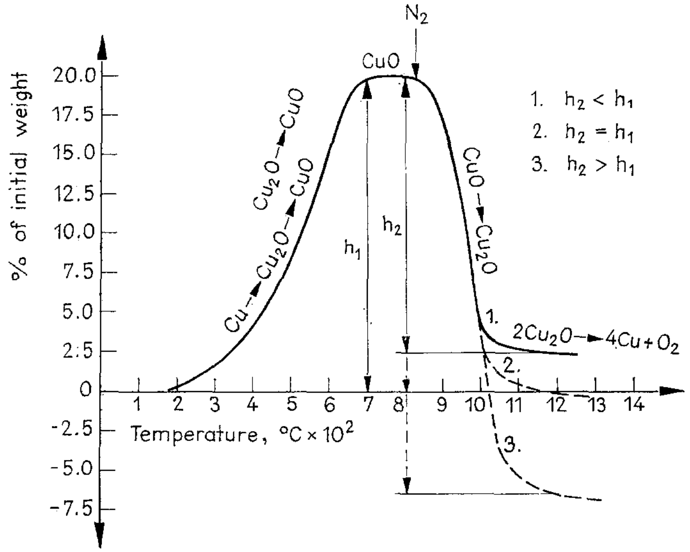

Fig. 8. TG curve of metallic copper, copper(I) oxide and copper(II) oxide

This heating curve may present three aspects:

1. $h_2 < h_1; \quad z < x$
2. $h_2 = h_1; \quad z = x$
3. $h_2 > h_1; \quad z > x$

As we can see, the shape of the heating curve is very similar to that of the binary mixture, Cu – CuO (Fig. 6).

Quantitative analysis

The quantitative determination of the unknown sample may be carried out by determining the size and the ratio of the two segments of the curve (h_1 and h_2):

(a) If the shape of the heating curve is similar to one of Figs 2, 3, 4, 7 respectively, then the material is:

metallic copper only	Fig. 2
– copper(I) oxide (Cu_2O)	Fig. 3
– copper(II) oxide (CuO)	Fig. 4
– copper(I) and copper(II) oxide (Cu_2O-CuO)	Fig. 7

(b) If the shape of the heating curve is similar to one of Figs 5, 6, 8 respectively, the components cannot be identified in this case without prior determination of the metallic copper content by another method.

The method which gives the best results in the presence of copper(I) and (copper(II) oxide is the Baker and Gibbs method [50, 51]. This method involves the following steps:

a – extraction of copper(I) oxide with alcohol and hydrochloric acid

b – reduction of copper(II) oxide with $SnCl_2$ followed by the same extraction with alcohol and hydrochloric acid, and

c – attack of metallic copper of the residue by $FeCl_3$, the resulting $FeCl_2$ (corresponding to metallic copper) being titrated by cerium sulphate.

The separation of small quantities of Cu_2O and CuO from Cu leads to appreciable losses of the Cu_2O and CuO, but the separation of metallic Cu is very accurate in this method.

Once the metallic copper content is known, we are able to determine the other components:

– if $h_1 = 2x$ and $h_2 > x$	metallic copper and copper(II) oxide are present (Fig. 6); $z = h_2 - x$; CuO $= 10z$;
– if $h_1 > 2x$ and $h_2 = x + y$	metallic copper and copper(I) oxide are present (Fig. 5); $y = h_1 - 2x$; $Cu_2O = 9y$;
– if $h_1 > 2x$ and $h_2 > x + y$	metallic copper, copper(I) and copper(II) oxides are present (Fig. 8); $y = h_1 - 2x$; $z = h_2 - x - y$; $Cu_2O = 9y$; CuO $= 10z$.

Examples of application

1. *Analysis of copper cement*

The copper cement was obtained as a thin metallic foil from a saturated solution of copper sulphate, acidified with sulphuric acid (one gram per liter) on the surface of a steel plate. The cement was washed with distilled water and acetone; its copper content was 99.88% Cu and 0.10% Fe.

After two days the microscopic examination of the cement in reflected and polarized light showed only metallic copper and copper(I) oxide.

The analysis of 1000 mg of cement indicates:

(a) 36 mg moisture (heating to 200° C and cooling to room temperature in purified nitrogen)

(b) $h_1 = 2x + y = 216$ mg

(c) $h_2 = x + y = 116.40$ mg

$x = h_1 - h_2 = 99.60$ mg ... metallic copper, Cu $= 8x = 796.80$ mg

$y = 16.80$ mg ... copper(I) oxide, $Cu_2O = 9y = 151.20$ mg

$H_2O + Cu + Cu_2O = 984$ mg instead of 1016.80 mg; $\Delta = 32.80$ mg $= 3.2\%$

2. *Analysis of mixture of copper oxides and metallic copper*

A synthetic mixture was prepared from:

- 100 mg metallic copper (fresh copper cement foil with 99.85% Cu)
- 675 mg copper(I) oxide, Cu_2O, prepared by heating copper(II) oxide in purified nitrogen to 1200° and cooling to room temperature
- 225 mg copper(II) oxide, CuO, "Fisher" reagent, with 0.33% H_2O.

The analysis indicates:

(a) 4.20 mg moisture

(b) $h_1 = 2x + y = 101.50$ mg

(c) $h_2 = x + y + z = 109.50$ mg

$x = 12.50$ mg (found by Baker and Gibbs method)

$y = 75.50$ mg ... copper(I) oxide, $Cu_2O = 9y = 679.50$ mg

$z = 21.50$ mg ... copper(II) oxide, $CuO = 10z = 215$ mg

Results

Components	Synthetic mixture, mg	Found by analysis, mg	Δ mg	± %
H_2O	0.74	0.42	—0.32	— 43
Cu, metallic	99.85	100.00	+0.15	+ 0.15
Cu_2O	675.00	679.50	+4.50	+ 0.66
CuO	224.20	215.00	—9.20	— 4.00
	999.79	994.92	—4.87	— 0.48

Summary and conclusions

Thermogravimetry can be used for the quantitative analysis of;

(a) mixtures of copper oxides:

(b) mixtures of metallic and copper oxides (the thermogravimetry must be accompanied by the determination of metallic copper by a wet method);

(c) oxidation process of copper, in the form of a metallic powder obtained by pyro-, hydro- or electrometallurgical processes.

The method cannot be applied to substances with other components undergoing weight changes by heating or those which may react with copper and its oxides.

*

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Résumé — Étude thermogravimétrique de mélanges du cuivre métallique et d'oxydes de cuivre. Les échantillons sont d'abord chauffés dans l'air jusqu'à 700—800° jusqu'à ce que tous les constituants soient transformés en oxyde de cuivre(II); le chauffage est ensuite poursuivi dans l'azote jusqu'à 1010—1100°, où la dissociation de l'oxyde de cuivre(II) en oxyde de cuivre(I) est complète. Les constituants ont été identifiés et dosés en utilisant la forme, la dimension et les proportions des différentes parties des courbes pendant le chauffage. La méthode peut être utilisée pour l'analyse quantitative de mélanges de cuivre et ou d'oxydes de cuivre.

Zusammenfassung. — Gemische von metallischem Kupfer und Kupferoxiden wurden thermogravimetrisch untersucht. Zur Überführung aller Komponenten in Kupfer(II)-oxid erhitzte man sie in Luft auf 700—800°, um daraufhin bis zur vollständigen Dissoziation des Kupfer(II)-oxids zu Kupfer(I)-oxid unter Stickstoff die Temperatur bis auf 1050—1100° zu steigern. Die Identifizierung der Komponenten und ihre quantitative Bestimmung erfolgten durch die Form, Größe und die Verhältnisse der verschiedenen Abschnitte der erhaltenen Kurven. Diese Methode ist zur quantitativen Bestimmung von Gemischen aus Kupfer und Kupferoxid sowie von Kupferoxiden geeignet.

Резюме — Исследованы смеси металлической и окисной меди методом термогравиметрии. Образцы нагревали в атмосфере воздуха до температуры 700—800: (при этом каждый компонент переходит в окись меди(II) ,и продолжали нагревание в азоте до 1050—1100°C [происходит диссоциация окиси меди(II) с образованием окиси меди(I)]. Идентифицированы и количественно определены компоненты путем установления вида, формата и соотношения сегментов кривых, полученных при нагревании. Метод применим для количественного анализа смесей меди и/или окисей меди.