Heraeus Quarzglas GmbH) over a measuring range from 190 nm -1,100 nm at a resolution of 2 nm.

The nuclear resonance spectroscopic measurements were carried out using a Bruker Avance 400 MHz NMR spectrometer (reference example 1) and a Bruker Avance 600 MHz NMR spectrometer (example, reference examples 2 and 3).

The platinum content was determined by gravimetry.

The educts used were platinum(IV) hydroxoacid ($H_2[Pt(OH)_6]$) from in-house production (w(Pt): 55.51 %), oxalic acid dihydrate for analysis EMSURE® ACS, ISO, Reag. Ph Eur made by Merck KGaA, art. no. 100495, and platinum oxalate from in-house production (w(Pt): 11.72 %).

Example 1 (Production of platinum oxalate at 40°C, in the presence of auto-catalyst, oxalic acid added in 5 aliquots)

A total of 10 g Pt (50 mmol), in the form of 18.01 g $H_2[Pt(OH)_6]$ were placed in 54.29 ml demineralised water ("VEW") in a 250 ml three-necked round flask.

Then, 0.04 g Pt oxalate (0.24 mmol Pt) were added as auto-catalyst at room temperature (23°C) while stirring (250 U/min) with a magnetic stirrer. A pale greenish suspension was thus produced.

Time: 0 min:
The suspension was heated in a water bath from room temperature to 40°C over the course of 20 minutes.

Time 20 min:
As soon as the temperature of the suspension had reached 40°C, one of five equal aliquots of 2.568 g (20 mmol) oxalic acid dihydrate each was added. Gas was observed to be produced instantaneously, which lasted for a period of 60 minutes. A total of 270 ml CO_2 were captured.

Time 80 min:
Once gas production ceased, another aliquot of 2.568 g (20 mmol) oxalic acid dihydrate was added. A total of 40 ml CO_2 were captured.

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Patent application

Heraeus Precious Metals GmbH & Co. KG

Method for the production of noble metal oxalate complexes

Patent claims

1. Method for the production of noble metal oxalate complexes, in which a noble metal precursor is reacted with oxalic acid and/or oxalic acid salt, characterised in that noble metal oxalate complexes corresponding to the product are added as an auto-catalyst.

2. Method for the production of noble metal oxalate complexes according to claim 1, characterised in that the reaction is carried out at a temperature below the decomposition temperature of the noble metal oxalate complexes.

3. Method for the production of noble metal oxalate complexes according to claim 2, characterised in that the reaction is carried out at a maximal temperature of 1°C below the decomposition temperature of the noble metal oxalate complexes.

4. Method for the production of noble metal oxalate complexes according to claim 1, characterised in that the reaction is carried out at a temperature between 0 and 56°C.

5. Method according to any one of the preceding claims, characterised in that
in the first step, an aqueous solution or suspension of noble metal precursor and noble metal oxalate complexes is produced,
in the second step, the aqueous solution or suspension from the first step is temperature-equilibrated to reaction temperature,
and,
in the third step, oxalic acid and/or oxalic acid salt is added.

6. Method according to any one of the claims 1 to 5, characterised in that the amount of auto-catalyst to be added is 1×10^{-4} to 5×10^{-2} molar equivalents of noble metal relative to the noble metal in the noble metal precursor solution.

7. Method according to any one of the claims 1 to 6, characterised in that oxalic acid and/or oxalic acid salt are selected from the group consisting of oxalic acid, sodium oxalate, ammonium oxalate, and potassium oxalate, and mixtures thereof.

8. Method according to any one of the preceding claims, characterised in that, the noble metal precursor is selected from the group consisting of noble metal oxide hydrate, noble metal nitrate, and noble metal acetate, and mixtures thereof.

9. Method according to any one of the claims 1 to 8, characterised in that the noble metal is platinum.

10. Method according to claim 9, characterised in that the platinum precursor is platinum oxide hydrate (platinum-(IV) hydroxoacid) or any of the salts thereof.

11. Method according to claim 9 or 10, characterised in that the reaction is carried out at a temperature from 30°C to 45°C.

12. Method according to any one of the claims 9 to 11, characterised in that 1.8 to 2.8 molar equivalents of oxalic acid relative to platinum in the platinum precursor are added.

13. Method according to any one of the claims 9 to 12, characterised in that
    1) a suspension of platinum oxide hydrate (platinum-(IV) hydroxoacid) in water is produced;
    2) a solution of platinum oxalate complexes in water is produced;
    3) the suspension from 1) and the solution from 2) are combined and the resulting mixture is temperature-equilibrated to reaction temperature;
    4) a first aliquot of 0.4 to 1.4 molar equivalents of oxalic acid, relative to platinum in platinum oxide hydrate (platinum-(IV) hydroxoacid), is added; and
    5) a second aliquot of 0.1 to 1.4 molar equivalents of oxalic acid, relative to platinum in platinum oxide hydrate (platinum-(IV) hydroxoacid), is added, and
    6) as an option, step 5) is repeated one or more times until a total amount of 1.8 to 2.8

molar equivalents of oxalic acid, relative to platinum in platinum oxide hydrate (platinum-(IV) hydroxoacid), has been added.

Heraeus Quarzglas GmbH) over a measuring range from 190 nm -1,100 nm at a resolution of 2 nm.

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Time 80 min:
Once gas production ceased, another aliquot of 2.568 g (20 mmol) oxalic acid dihydrate was added. A total of 40 ml CO_2 were captured.

Claims

1. Method for the production of platinum oxalate complexes, in which a platinum precursor is reacted with oxalic acid and/or oxalic acid salt, wherein platinum oxalate complexes corresponding to the product are added as an auto-catalyst.

2. Method for the production of platinum oxalate complexes according to claim 1, wherein the reaction is carried out at a temperature below the decomposition temperature of the platinum oxalate complexes.

3. Method for the production of platinum oxalate complexes according to claim 2, wherein the reaction is carried out at a maximal temperature of 1°C below the decomposition temperature of the platinum oxalate complexes.

4. Method for the production of platinum oxalate complexes according to claim 1, wherein the reaction is carried out at a temperature between 0 and 56°C.

5. Method according to any one of the preceding claims, wherein in the first step, an aqueous solution or suspension of platinum precursor and platinum oxalate complexes is produced,
in the second step, the aqueous solution or suspension from the first step is temperature-equilibrated to reaction temperature,
and,
in the third step, oxalic acid and/or oxalic acid salt is added.

6. Method according to any one of the claims 1 to 5, wherein the amount of auto-catalyst to be added is 1×10-4 to 5×10-2 molar equivalents of platinum relative to the platinum in the platinum solution.

7. Method according to any one of the claims 1 to 6, wherein oxalic acid and/or oxalic acid salt are selected from the group consisting of oxalic acid, sodium oxalate, ammonium oxalate, and potassium oxalate, and mixtures thereof.

8. Method according to any one of the preceding claims, wherein, the platinum precursor is selected from the group consisting of platinum oxide hydrate, platinum nitrate, and platinum acetate, and mixtures thereof.

9. Method according to claim 1, wherein the platinum precursor is platinum oxide hydrate (platinum-(IV) hydroxoacid) or any of the salts thereof.

10. Method according to claim 1 or claim 9, wherein the reaction is carried out at a temperature from 30°C to 45°C.

11. Method according to any one of the claims 1 to 10, wherein 1.8 to 2.8 molar equivalents of oxalic acid relative to platinum in the platinum precursor are added.

12. Method according to any one of the claims 1 to 11, wherein
1) a suspension of platinum oxide hydrate (platinum-(IV) hydroxoacid) in water is produced;
2) a solution of platinum oxalate complexes in water is produced;
3) the suspension from 1) and the solution from 2) are combined and the resulting mixture is temperature-equilibrated to reaction temperature;
4) a first aliquot of 0.4 to 1.4 molar equivalents of oxalic acid, relative to platinum in platinum oxide hydrate (platinum-(IV) hydroxoacid), is added; and
5) a second aliquot of 0.1 to 1.4 molar equivalents of oxalic acid, relative to platinum in platinum oxide hydrate (platinum-(IV) hydroxoacid), is added, and
6) as an option, step 5) is repeated one or more times until a total amount of 1.8 to 2.8 molar equivalents of oxalic acid, relative to platinum in platinum oxide hydrate (platinum-(IV) hydroxoacid), has been added.