606492

COMMONWEALTH OF AUSTRALIA

Patents Act 1952

# CONVENTION APPLICATION FOR A STANDARD PATENT

~~I~~/WE, SKW TROSTBERG AKTIENGESELLSCHAFT, a West German corporation of Dr.-Albert-Frank Strasse 32, D-8223 Trostberg Federal Republic of Germany
hereby apply for the grant of a Standard Patent for an invention entitled:
AGENT AND PROCESS FOR OBTAINING NOBLE METALS FROM THIOUREA COMPLEXES THEREOF

which is described in the accompanying complete specification.

This application is made under the provision of Part XVl of the Patents Act 1952 and is based on an application for a patent or similar protection made

in Federal Republic of Germany on 19 January 1988
No. (P38 01 340.1)

~~in~~ ~~on~~
~~No.~~ (

~~My~~/Our address for service is:

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28A Montague St.
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Dated this 5th day of January 1989,
SKW TROSTBERG AKTIENGESELLSCHAFT

By: 
Registered Patent Attorney

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SKW 959

# Commonwealth of Australia
## The Patents Act 1952
# DECLARATION IN SUPPORT

In support of the (Convention) Application made by:

SKW TROSTBERG AKTIENGESELLSCHAFT, a West German corporation of Dr.-Albert-Frank-Strasse 32, D-8223 Trostberg, Federal Republic of Germany

for a patent for an invention entitled:

AGENT AND PROCESS FOR OBTAINING NOBLE METALS FROM THIOUREA COMPLEXES THEREOF

~~I~~ (We) Dr. Rock and Dr. Prietzel

of and care of the applicant company do solemnly and sincerely declare as follows:

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~~a) I am (We are) the applicant(s) for the patent~~

~~or~~

b) ~~I am~~ (We are) authorised by the applicant~~(s)~~ for the patent to make this declaration on its behalf.

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Delete the following if not a Convention Application.

The basic application~~(s)~~ as defined by section 141 (1~~a~~) of the Act was ~~(were)~~ made

in Federal Republic of Germany on 19 January 1988

~~in~~ ~~on~~

~~in~~ ~~on~~

by the present applicant company

The basic application~~(s)~~ referred to in this paragraph is ~~(are)~~ the first application~~(s)~~ made in a Convention country in respect of the invention the subject of the application.

---

~~a) I am (We are) the actual inventor(s) of the invention.~~

~~or~~

b) Dr. Reinhold Schulze, Baumburger Leite 2, D-8226 Altenmarkt
Federal Republic of Germany

Is ~~(are)~~ the actual inventor~~(s)~~ of the invention and the facts upon which the applicant company is ~~(are)~~ entitled to make the application are as follows:

the applicant is a person who would if a patent were granted upon an application made by the actual inventor, be entitled to have the patent assigned to it.

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Declared at Trostberg this 1st day of December 1988

Signed ........ Status ........

SKW Trostberg Aktiengesellschaft

Declarant's Name ........

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Vorstand

Dr. Prietzel
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F. B. RICE & CO PATENT ATTORNEYS

This form is suitable for any type of Patent Application. No legalisation required.

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(56) Prior Art Documents
AU 566716 39219/85 C22B 11/04
AU 560861 34738/84 C22B 11/04
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(57) Claim

1. Agent for the elution of thiourea-noble metal complexes from active charcoal based on an acidic aqueous solution containing thiourea and an organic solvent, wherein it has a content of 0.1 to 50g/litre of thiourea and contains at least one organic solvent which is partly soluble in water.

6. A process for obtaining noble metals from thiourea-noble metal complex absorbed on active charcoal comprising the following reaction steps:

a) eluting the thiourea-noble metal complex from the active charcoal at ambient or elevated temperature with 1 to 100ml of an agent according to any of claims 1 to 5 per 0.1 gram of loaded active charcoal and

.../2

b) precipitating the noble metals from the aqueous organic phase by the addition of base metals in the presence of a small amount of sulphite and optionally reactivating the active charcoal with an aqueous solution of a sulphite.

10. Process according to any of claims 6 to 9, wherein the active charcoal is reactivated by reaction with a solution which contains 1 to 100g of sulphite/litre at a temperature of 50 to 95°C for 0.15 to 4 hours.

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# COMPLETE SPECIFICATION

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Complete Specification for the invention entitled:
AGENT AND PROCESS FOR OBTAINING NOBLE METALS FROM THIOUREA COMPLEXES THEREOF

The following statement is a full description of this invention including the best method of performing it known to us/~~me~~:-

The present invention is concerned with an agent and a process for obtaining noble metals from thiourea complexes thereof adsorbed on active charcoal.

A special form of use for obtaining noble metal complexes adsorbed on active charcoal is the so-called carbon-in-pulp process which has achieved importance on a large scale. In the case of this process, granulated active charcoal of about 0.5 mm. grain diameter or above is added directly to a finely ground suspension of ore or of other raw materials containing noble metal, such as roasting residues and "lyes" obtained after completion of the dissolving of noble metals. A laborious separation of the leached out solid material from the noble metal-containing liquid is thereby avoided since the noble metal-containing active charcoal can be separated off in a simple manner by coarse-meshed sieves from the useless leaching residues.

The noble metals must be dissolved from the active charcoal in a further process step. In the case of the use of cyanides as leaching agents, the noble metals are present on the active charcoal as adsorbed cyanide complexes. For the desorption of the noble metal, the active charcoal must be treated with alkaline cyanide solution preferably under pressure at temperatures above 100°C. This process is technically laborious since, in particular, it requires working with the highly toxic cyanides.

The use of thiourea as leaching agent for noble metals, which is substantially less harmful in comparison with the cyanides, has also been known for a long time. Furthermore, in comparison with cyanides, thiourea has a higher speed of dissolving of noble metals and the noble metal complexes thereof are adsorbed more quickly and to a greater extent by active charcoal.

In order again to be able to elute thiourea-noble metal complexes from the active charcoal, there has been used the laborious pressure technique known from cyanide leaching. According to another suggestion, elution is carried out with strongly acidic (50 g./ litre sulphuric acid), highly concentrated thiourea solutions (100 g./litre thiourea). Since, however, the noble metals cannot be obtained directly from these strongly acidic solutions, a preceding neutralisation with a considerable expense for neutralisation chemicals and a further filtration are necessary.

The use of alcohol- or glycol-containing solutions, such as have been suggested for the elution of cyanide-adsorbed noble metals, does not give the desired result in the case of thiourea-noble metal complexes (see Engineering & Mining Journal, June, 1987, pp. 48, 49).

Therefore, it is an object of the present invention to provide a technically simple and economic process for the elution of thiourea-noble metal complexes adsorbed on active charcoal and to obtain the noble metals therefrom in metallic form.

Thus, according to the present invention, there is provided an agent for the elution of thiourea-noble metal complexes from active charcoal based on an acidic aqueous solution containing thiourea and an organic solvent, wherein it has a content of 0.1 to 50 g./litre thiourea and contains at least one organic solvent which is partly soluble in water.

The present invention also provides a process for obtaining noble metals using the agent according to the present invention which comprises the following reaction steps:

a) elution of the thiourea-noble metal complex from the active charcoal at ambient or elevated temperature with 1 to 100 ml. of the agent according to the present invention per 0.1 gram of loaded active charcoal and

b) precipitation of the noble metals from the aqueous organic phase by the addition of base metals in the presence of a small amount of sulphite and optional reactivation of the active charcoal with an aqueous solution of a sulphite.

Surprisingly, this agent and process permit, under relatively mild conditions and with high selectivity, the obtaining of the noble metals silver and gold from finely ground ore slurries. Furthermore, in comparison with older processes, working is carried out with solutions of relatively low concentrations so that losses of chemicals and organic solvents remain small.

As organic solvents which are partly soluble in water, there can be used, in particular, mono- and polyhydroxy alcohols, substituted alcohols, esters, ethers (also cyclic ethers, such as dioxan or tetrahydrofuran), ketones, nitriles, acid amides (see the following Table 6).

A preferred agent is an aqueous solution, adjusted to a pH value of 1 to 3, with contents of 1 to 50 g./litre of thiourea and 0.1 to 20% by volume of at least one alcohol containing 3 to 5 carbon atoms, of a glycol or of a glycol ether. Especially preferred is an aqueous solution which has a pH value of 1.5 to 2.5 adjusted by means of an acid and which contains 1 to 50 g./litre thiourea and 0.1 to 10% by volume of n-butanol (see the following Table 2).

The combination according to the present invention of thiourea and organic solvent partly soluble in water leads to a surprising synergistic effect in the case of the elution of the noble metal from the active charcoal since, under the chosen conditions, neither an acidifed thiourea solution nor an acidified solution containing, for example, n-butanol alone brings about a satisfactory noble metal elution. Only the combination in the form of an acidified aqueous thiourea solution with a content of organic solvent leads, unexpectedly, to a high elution of noble metal (see the following Table 1).

The process according to the present invention for the elution of thiourea-noble metal complexes adsorbed on active charcoal is carried out with the agent according to the present invention preferably at ambient temperature or at a temperature of up to 95°C., there thereby being used 1 to 100 ml. of elution solution per 0.1 g. of loaded active charcoal, the thiourea concentration of which is from 0.1 to 50 g./litre, the content of organic solvent partly soluble in water being from 0.1 to 20% by volume and the pH value of which has been adjusted by acid to a value of from 1 to 5.

When the elution of the thiourea-noble metal complex is finished, the noble metal is advantageously precipitated out from the aqueous organic phase, after separating off the active charcoal, by the addition of base metals. It is thereby preferred to work in the presence of a small amount of sulphite and optionally to regenerate the active charcoal with an aqueous sulphite solution.

Preferred conditions for the process are: elution at a temperature of from 20 to 80°C. (see the following Table 3); concentration of 1 to 10 g. of thiourea/litre in the elution solution; content of organic solvent of from 1 to 20% by volume; pH value of the solution 1.5 to 3, preferably by the addition of sulphuric acid. Especially preferably, as organic solvents which are partly soluble in water, there are used propanols, butanols, amyl alcohols or glycol ethers. These solvents can be used alone or in the form of mixtures. In the following Table 6, there are listed a number of organic solvents which are partly soluble in water which, in combination with an aqueous, acidic thiourea solution (10 g. of thiourea/litre; pH 2), give a distinct synergistic effect in the case of the dissolving off of silver and/or gold complexes from active charcoal.

As active charcoals, there can be used commercially available products. These must merely be solid enough in aqueous solutions in order that they can be filtered off practically completely from the fine dispersed slurry. Especially suitable active charcoals are provided, inter alia, by the firms Calgon (for example Calgon GRC 22), Haycarb Carbon 6 - 16, Le Carbon (for example PICA G 210), Degussa (for example Degusorb C 25), Norite (for example R 2520) and Kop Ank, 8 - 16.

An especial advantage of the present invention is to be seen in the fact that the elution is very selective for noble metals adsorbed on active charcoal. Simultaneously co-adsorbed other metals, such as especially copper, lead or other elements, such as sulphur, arsenic, are dissolved off from the charcoal by the elution agent either in an insignificant amount or not at all. The noble metals can thereby be obtained in high purity from the eluate, for example by cementation with finely pulverised base metals, such as iron, aluminium, magnesium or zinc. It is preferred to use the inexpensive iron. To the precipitation solution is added a small amount of sulphite, for example 0.05 to 5 g./litre, in the form of sulphur dioxide or sodium sulphite, it being preferred to use sodium pyrosulphite in an amount of about 0.1 to 1 g./litre. The sulphite addition increases, on the one hand, the yield of the noble metal or, on the other hand, lowers the base metal requirement for the cementation (see the following table 4). The cementation preferably takes place by the addition of 1 to 10 weight units of base metal per weight unit of complex-bound noble metal in the case of a content of 0.1 to 1 g./litre of sulphite in the reaction solution at a temperature of from 40 to 70°C. It is substantially complete within a period of from 0.15 to 4 hours.

Since, in the case of repeated loading and discharge steps, the charcoal can become increasingly inactive, it is advantageous continuously to regenerate this, i.e. to free it from still adsorbed impurities, in order to retain its original loading capacity. A process which has proved to be suitable for this purpose is a treatment with sulphites, for example sodium sulphite, sodium pyrosulphite or sodium hydrogen sulphite, at an elevated temperature. The contaminated active charcoal is preferably regenerated at a temperature of from 50 to 95°C. for a period of from 0.15 to 4 hours with an aqueous solution containing 1 to

100 g./litre of sodium sulphite. Thereafter, the active charcoal again possesses its original adsorption capacity for noble metal-thiourea complexes (see the following Table 5).

The following Examples are given for the purpose of illustrating the present invention:

Example 1.

Elution of silver from active charcoal with acidified thiourea solution, acidified butanol-containing solution and acidified butanol-containing thiourea solution.

For the demonstration of the synergistic effect in the case of the dissolving off of silver from active charcoal, there was used an active charcoal of the Calgon GRC 22 type. After a leaching process with thiourea, this contained 40% of water and 3% of silver in the dry material.

50 ml. amounts of solutions 1 to 3 (see the following Table 1) were shaken with 0.1 g. of active charcoal Calgon GRC 22 in a waterbath at 70°C. for 3 hours and subsequently analysed for eluted silver.

As can be seen from the following Table 1, with an acidified butanol-containing thiourea solution, practically all of the silver is dissolved out from the charcoal, whereas with a solution containing only butanol or only thiourea, only an unsatisfactory amount of the silver is obtained.

Table 1

| | solution 1 | solution 2 | solution 3 |
|---|---|---|---|
| thiourea g/l. | 10 | - | 10 |
| n-butanol ml./l. | - | 50 | 50 |
| sulphuric acid g./l. (pH 2) | 0.66 | 0.66 | 0.66 |
| eluted silver mg./l. | 4.3 | 1.6 | 35.2 |
| eluted silver % | 11.9 | 4.5 | 92.9 |

Example 2.

Influence of the concentration of thiourea and of the content of butanol in the elution solution on the elution of silver.

The experimental conditions were analogous to those described in Example 1. Active charcoal of the Calgon GRC 22 type was used which is loaded with 0.52% by weight of silver. Elution was carried out by shaking for 3 hours in a waterbath at 70°C. with increasing amounts of butanol and increasing amounts of thiourea in the elution agent.

The results obtained are set out in the following Table 2:

Table 2

| 1-butanol ml./l. | thiourea g./l. | eluted silver (%) |
|---|---|---|
| 10 | - | 2.6 |
| 10 | 2 | 32.9 |
| 10 | 5 | 37.0 |
| 10 | 10 | 39.0 |
| 20 | - | 3.8 |
| 20 | 2 | 58.6 |
| 20 | 5 | 58.9 |
| 20 | 10 | 62.7 |
| 30 | - | 5.1 |
| 30 | 2 | 69.9 |
| 30 | 5 | 68.0 |
| 30 | 10 | 79.1 |
| 50 | - | 4.5 |
| 50 | 2 | 95.0 |
| 50 | 5 | 96.0 |
| 50 | 10 | 97.8 |

### Example 3.

Influence of the temperature on the elution of silver.

The experimental conditions corresponded to those of Example 1. An active charcoal of the firm Degussa Degusorb C 25 (as dry substance) loaded with 2.8% silver was eluted by shaking for 3 hours at different temperatures with a solution which contained 50 ml./

litre butanol and 10 g./litre thiourea and which had been acidified with sulphuric acid to pH 2.

The results obtained are set out in the following Table 3:

Table 3

| temperature (°C.) | eluted silver (%) |
|---|---|
| 20 | 59.6 |
| 40 | 72.0 |
| 60 | 94.5 |
| 70 | 96.0 |
| 80 | 97.5 |

Example 4.

Cementation of silver from the elution solution.

As starting solution, there was used an elution solution of pH 2 containing 5% by volume of butanol, 10 g./litre of thiourea and 99 mg./litre of silver.

The cementation took place by the addition of 250 mg./litre of iron powder and increasing additions of sodium pyrosulphite at 60°C. during the course of 2 hours.

The results obtained are set out in the following Table 4:

### Table 4

| experiment No. | iron powder mg./l. | $Na_2S_2O_5$ mg./l. | dissolved silver mg./l. | $\frac{Fe}{\Delta Ag}$ |
|---|---|---|---|---|
| 1 | - | - | 99 | - |
| 2 | 250 | - | 52 | 5.32 |
| 3 | 250 | 250 | 44 | 4.55 |
| 4 | 250 | 500 | 35 | 3.91 |
| 5 | 250 | 750 | 19 | 3.13 |
| 6 | - | 750 | 98 | - |

$$\frac{Fe}{\Delta Ag} = \frac{\text{amount of iron powder used}}{\text{amount of precipitated silver}}$$

### Example 5.

Regeneration of the active charcoal by treatment with sodium sulphite solutions.

There was used the active charcoal PICA G 210 8-16 which had served for the adsorption of silver from a leaching process with thiourea from zinc roasted material.

After elution of the silver with acidic butanol/ thiourea solution, the sieved off and washed charcoal contained 25% by weight moisture.

25 g. amounts of this charcoal were treated in 1 litre of water with increasing amounts of sodium sulphite for 3 hours at 70°C. and subsequently again used for the silver adsorption.

The following Table 5 shows the results which were obtained with a regenerated active charcoal which had been treated with increasing amounts of sodium sulphite and were used for obtaining silver from an elution solution containing 350 mg./litre silver.

Table 5

| experiment No. | active charcoal g/l. | $Na_2SO_3$ g./l. | silver remaining in the solution mg./l. |
|---|---|---|---|
| 1 | 25 | - | 48 |
| 2 | 25 | 2.5 | 24 |
| 3 | 25 | 5.0 | 9 |
| 4 | 25 | 50.0 | 8 |

Example 6.

Elution of silver and/or gold complexes of thiourea from active charcoal with different organic solvents.

Experimental conditions:

In each case, there was eluted:

a) 1 g. active charcoal with 40% by weight moisture and 0.52% by weight silver in the dry substance;

b) 0.5 active charcoal with 40% by weight moisture and 2.55% by weight gold in the dry substance,

with 40 ml. of an aqueous solution which contained 10 g./litre thiourea, 0.66 g./litre sulphuric acid (pH 2) and in each case 2 ml. of one of the organic solvents mentioned in the following Table 6 (in the case of experiments 7 to 10, only 1 ml. of the organic solvent was added).

The period of elution was 3 hours at 70°C. in a shaking waterbath.

Since the desorption of silver and gold complexes of thiourea are substantially analogous, in the case of the gold elution, not all of the solvents were investigated. In the following Table 6, there are shown the results obtained with the various solvents. A dash means that an experiment was not carried out.

Table 6

| experiment No. | organic solvent | % silver eluted | % gold eluted |
|---|---|---|---|
| | base solution without additive | 4.8 | 5.3 |
| 1 | ethanol | 27.5 | 41.2 |
| 2 | *n*-propanol | 69.3 | 78.2 |
| 3 | isopropanol | 60.7 | 71.4 |
| 4 | *n*-butanol | 94.5 | 90.1 |
| 5 | isobutanol | 82.6 | 90.9 |
| 6 | *tert*.-butanol | 61.8 | 78.3 |
| 7 | *n*-amyl alcohol | 89.7 | 83.6 |
| 8 | isoamyl alcohol | 95.1 | 83.6 |
| 9 | *tert*.-amyl alcohol | 80.2 | 79.6 |
| 10 | benzyl alcohol | 89.7 | - |
| 11 | methoxybutanol | 61.2 | 80.6 |
| 12 | propane-1,3-diol | 27.5 | - |
| 13 | butane-1,2-diol | 42.9 | - |
| 14 | butane-1,3-diol | 31.7 | - |

Table 6 (cont.)

| | | | |
|---|---|---|---|
| 15 | butane-1,4-diol | 22.7 | - |
| 16 | butane-2,3-diol | 22.9 | - |
| 17 | 2-ethylhexane-1,3-diol | 80.2 | - |
| 18 | ethyleneglycol | 8.6 | - |
| 19 | diethyleneglycol | 27.8 | 40.6 |
| 20 | triethyleneglycol | 46.2 | - |
| 21 | polyethyleneglycol 400 | 62.5 | - |
| 22 | dipropyleneglycol | 64.6 | 72.6 |
| 23 | methylglycol | 26.5 | - |
| 24 | methyldiglycol | 43.5 | - |
| 25 | ethylglycol | - | 71.6 |
| 26 | butylglycol | 73.4 | - |
| 27 | butyldiglycol | 74.7 | - |
| 28 | methylglycol acetate | 69.3 | 95.5 |
| 29 | butyldiglycol acetate | 73.4 | - |
| 30 | diethyleneglycol dimethyl ether | 77.5 | 80.6 |
| 31 | tetrahydrofuran | 74.5 | 79.8 |
| 32 | 1,4-dioxan | 50.3 | - |
| 33 | methyl acetate | 62.5 | - |
| 34 | ethyl acetate | 72.0 | 82.6 |
| 35 | acetonitrile | 27.9 | 36.2 |
| 36 | dimethyl sulphoxide | 13.3 | - |
| 37 | dimethylformamide | 26.5 | - |
| 38 | formamide | 7.2 | - |
| 39 | methyl isobutyl ketone | 77.5 | - |
| 40 | diacetone alcohol | - | 84.6 |

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. Agent for the elution of thiourea-noble metal complexes from active charcoal based on an acidic aqueous solution containing thiourea and an organic solvent, wherein it has a content of 0.1 to 50g/litre of thiourea and contains at least one organic solvent which is partly soluble in water.

2. Agent according to claim 1, wherein the organic solvent which is partly soluble in water is a mono- or polyhydroxy optionally substituted alcohol, ether, ester or ketone.

3. Agent according to claim 1 or 2, wherein it has a pH value of from 1 to 5 and contains 1 to 50g/litre of thiourea and 0.1 to 20% by volume of an alcohol containing 3 to 5 carbon atoms.

4. Agent according to claim 3, wherein it has a pH value of from 1.5 to 2.5 adjusted by means of an acid and contains 0.1 to 10% by volume of *n*-butanol.

5. Agent according to claim 1 for the elution of thiourea-noble metal complexes from active charcoal, substantially as hereinbefore described and exemplified.

6. A process for obtaining noble metals from thiourea-noble metal complex absorbed on active charcoal comprising the following reaction steps:

a) eluting the thiourea-noble metal complex from the active charcoal at ambient or elevated temperature with 1 to 100ml of an agent according to any of claims 1 to 5 per 0.1 gram of loaded active charcoal and

b) precipitating the noble metals from the aqueous organic phase by the addition of base metals in the presence of a small amount of sulphite and optionally reactivating the active charcoal with an aqueous solution of a sulphite.

7. Process according to claim 6, wherein the elution of the noble metal-thiourea complex is carried out at a temperature of from 20 to 80°C, the concentration of thiourea in the solution is adjusted to 1 to 10g/litre and that of the organic solvent to 1 to 20% by volume and the pH value of the solution is adjusted to 1.5 to 3.

8. Process according to claim 6 or 7, wherein for the precipitation of the noble metal, per unit weight of complex-bound noble metal there are added 1 to 10 units of iron in pulverised form, as well as 0.1 to 1g of sulphite/litre of reaction solution.

9. Process according to claim 8, wherein the sulphite is added in the form of sodium sulphite.

10. Process according to any of claims 6 to 9, wherein the active charcoal is reactivated by reaction with a solution which contains 1 to 100g of sulphite/litre at a temperature of 50 to 95°C for 0.15 to 4 hours.

11. Process according to claim 6 for obtaining noble metals, substantially as hereinbefore described and exemplified.

12. Noble metals, whenever obtained by the process according to any of claims 6 to 11.

Dated this 5th day of January 1989

SKW TROSTBERG AKTIENGESELLSCHAFT
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