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(71) Applicants:

- **RESEARCH INSTITUTE FOR PRODUCTION DEVELOPMENT**
Kyoto-shi, Kyoto-hu 606 (JP)
- **MITSUI & CO., LTD.**
Chiyoda-ku Tokyo 100 (JP)
- **NEOS CO., LTD.,**
Kobe-shi, Hyogo-ken 650 (JP)

(72) Inventors:

- **TANIMOTO, Fumio**
Research Inst.for Production Dev.
KKyoto-shi, Kyoto-hu 606 (JP)
- **TSUKUMO, Kiyohiko**
Research Inst.for Prod. Dev.
Kyoto-shi, Kyoto-hu 606 (JP)

• **NAKAMURA, Atsuhiko**

Kashiwa-shi Chiba-ken 277 (JP)

• **YANO, Tsuneo**

Hanno-shi Saitama-ken 357 (JP)

• **SUZUKI, Nobuhiro**

Tokyo Electric Power Company

Yokohama-shi Kanagawa-ken 230 (JP)

• **TOMA, Toshinori**

Tokyo Electric Power Company

Yokohama-shi Kanagawa-ken 230 (JP)

• **FURUHASHI, Koichi**

Kohga-gun Shiga-ken 520-32 (JP)

• **NAKAYAMA, Nobuyuki**

Kohga-gun Shiga-ken 520-32 (JP)

(74) Representative: **Klunker . Schmitt-Nilson . Hirsch**

Winzererstrasse 106

80797 München (DE)

(54) **METHOD OF DECOMPOSING HALOGENATED AROMATIC COMPOUND WITH ALKALINE SUBSTANCE**

(57) In the method of decomposing halogenated aromatic compounds, halogenated aromatic compounds and alkalis are contacted in a non-proton polar solvent at a temperature ranging from about 150 ° C to about 300° C for about 1 to about 10 hours. The blending ratio of alkalis with respect to the whole reaction system when the reaction starts is set at 5,000 mg/kg or more. Whereby, the halogenated aromatic compounds can be decomposed certainly and safely.

EP 0 711 580 A1

Description

Technical Field

5 The present invention relates to a safe method for decomposing halogenated aromatic compounds such as polychlorinated biphenyl (hereinafter "PCB") using alkalis in a polar solvent.

Background Art

10 Since it is extremely difficult to treat halogenated aromatic compounds such as PCB, considerable efforts have been made to remove or decompose halogenated aromatic compounds for more than twenty years. Methods for accomplishing this using a reaction process that takes place in the presence of an alkali include the alumina-alkali process disclosed by U.S. Patent No. 2,951,804. U.S. Patent No. 4,532,028 discloses a method of reacting alkali and a PCB content of up to 50,000 ppm at a temperature of 200° C or below in a mixture of alkyl or alkylene sulfoxide and polyole, thereby reducing
 15 the content to several ppm. Other examples include Canadian Patent No. 1,181,771 which discloses a method employing melted sodium, and Italian Patent No. 1,206,508 which disclose a method using alkaline earth metal on which polyethylene glycol is adsorbed.

Though each of the prior art techniques has its good points, it is not possible to further remove halogenated aromatic compounds from samples having a low concentration thereof, so that the halogenated aromatic compound content is
 20 further reduced to the extent that the inclusion thereof is substantially not recognizable; it is not yet possible to reduce the halogenated aromatic compound concentration to 1 ppm or below. In addition, heating the solvent used in the prior art methods to a high temperature of 150° C or over in the presence of an alkali or alkali metal has a chemically destabilizing effect that promotes solvent decomposition and polymerization, therefore these methods cannot be carried out industrially.

25 Furthermore, a reduction method using paraffin as a hydrogen source which has been studied recently is also defective in that about 1 ppm of PCB remains and the reaction temperature is high.

Disclosure of Invention

30 The inventor of the present invention investigated various ways of eliminating such drawbacks and discovered a highly effective method of decomposing halogenated aromatic compounds. In accordance with the method, in a non-proton polar solvent which has a high boiling point and good high-temperature stability with respect to alkalis, halogenated aromatic compounds are contacted at a high temperature with such an amount of alkalis that cannot be dissolved into the non-proton solvent.

35 Thus, in the method of the present invention for decomposing halogenated aromatic compounds by contacting halogenated aromatic compounds with alkalis in a non-proton polar solvent, the contact is carried out at a temperature ranging from about 150° C to about 300° C, for 1 to 10 hours, and the blending ratio of alkalis to the whole reaction system when the reaction starts is 5,000 mg/kg or more.

In particular, it is preferred that the blending ratio of alkalis to the whole reaction system when the reaction starts
 40 be 7,000 mg/kg or more.

The halogenated aromatic compounds to be decomposed include one halogenated aromatic compound selected from a group consisting of polychlorinated biphenyl, polychlorinated terphenyl, polybrominated biphenyl, and analogous compounds thereof, or a mixture of two or more halogenated aromatic compounds selected from this group.

With respect to the method in accordance with the present invention, it is not yet possible to explain theoretically as
 45 an essential reaction mechanism through what chemical reactions halogenated aromatic compounds are decomposed. After repeatedly investigating the decomposition method in accordance with the present invention, it is confirmed that halogenated aromatic compounds are decomposed to such an extent that halogenated aromatic compounds are substantially undetectable. The method is generally used in which, of chemicals that react, a less expensive chemical is added excessively to complete the chemical reaction. In the method of the present invention, however, the amount of
 50 alkalis is determined depending on the amount of the non-proton polar solvent serving as a solvent. That is to say, according to the present invention, it is to be noted that the action of the non-proton polar solvent is promoted by making the amount of the alkalis larger than that of the non-proton polar solvent rather than by making the amount of the alkalis larger than that of the halogenated aromatic compounds.

In the present invention, whether the starting material is 100 % halogenated aromatic compounds or halogenated
 55 aromatic compounds diluted to several ppm, it was confirmed that the halogenated aromatic compounds are decomposed to such an extent that the halogenated aromatic compounds are substantially undetectable. Therefore, according to the present invention, it is possible to treat not only 100 % halogenated aromatic compounds but also halogenated aromatic compounds diluted to the concentration ranging from 2 ppm to 80 % by hydrocarbon oil, for example, the principal component of which is non-aromatic hydrocarbon.

In the method in accordance with the present invention, as alkalis, one alkali selected from a group consisting of sodium hydroxide (NaOH), potassium hydroxide (KOH), calcium hydroxide ($\text{Ca}(\text{OH})_2$) and magnesium hydroxide ($\text{Mg}(\text{OH})_2$), or a mixture of two or more alkalis selected from this group can be used. Calcium hydroxide ($\text{Ca}(\text{OH})_2$) and others may be added to the reaction system in the form of the alkalis being oxides such as calcium oxide (CaO).

According to the present invention, there are found to be slight differences in the halogenated aromatic compound decomposition effect of the various non-proton polar solvents. Under any conditions, 1,3-dimethyl-2-imidazolidinone, tetramethylene sulfone, or a mixture of 1,3-dimethyl-2-imidazolidinone and tetramethylene sulfone are effective non-proton polar solvents.

Industrially these non-proton polar solvents are used relatively extensively, and the solvents are marketed and easily available. It is to be noted that these non-proton polar solvents can dissolve a large quantity of halogenated aromatic compounds as well as the solvents have low toxicity and risk. In the prior art techniques, it was clear that when the amount of halogenated aromatic compounds was decreased to, for example, in the order of parts per million, the reaction speed of halogenated aromatic compounds and alkalis remarkably lowered. When the above-mentioned non-proton polar solvents were used, it was confirmed that halogenated aromatic compounds were decomposed to the concentration of several ppm, and further to below the detection limit value (0.5 ppb or less), and that the compounds were substantially eliminated.

As non-proton polar solvents, a mixture the principal component of which is a solvent selected from a group consisting of 1,3-dimethyl-2-imidazolidinone (hereinafter DMI), tetramethylene sulfone, and a mixture of 1,3-dimethyl-2-imidazolidinone and tetramethylene sulfone, and which contains one polar solvent selected from dimethyl sulfoxide, N-methyl pyrrolidone, tetramethyl urea, diethylene glycol and polyethylene glycol dimethyl ether, or two or more polar solvents selected from this group at a concentration of 35 % or less, can be used to effectively decompose the halogenated aromatic compounds.

Best Mode for Carrying Out the Invention

Table 1 shows the treatment conditions and the remaining PCB amount after the treatment in the examples according to the present invention and the comparative examples. Note that in Table 1 when the remaining PCB amount is below the detection limit, it is represented by "N.D." in the column of the remaining PCB amount.

Example 1

As listed in Table 1, after 50 g of insulating oil (hydrocarbon oil the principal component of which is non-aromatic hydrocarbon) containing about 80 ppm of PCB, 100 g of DMI and 2 g of powdery NaOH were mixed in a flask, the mixture was stirred briskly while being maintained at a temperature of 200° C for about 6 hours. After cooling the mixture to room temperature, the lower layer of DMI was removed. Then the PCB in the oil layer was analyzed by a gas chromatography mechanical spectrometer (hereinafter GC-MS), whereby it was confirmed that the PCB content had decreased to below the detection limit value of the GC-MS, that is, 0.5 ppb (0.5 $\mu\text{g/kg}$) or less. Under the conditions of this example, the initial blending ratio of alkalis with respect to the whole reaction system corresponds to 13,000 mg/kg, and the ratio of alkalis with respect to the chlorine amount in the PCB (the number of moles of alkalis / the number of moles of chlorine) corresponds to 800 in terms of mole ratio.

Note that after the treatment was carried out for about 2 hours under the same conditions, it was confirmed that the concentration of the PCB in the oil layer had decreased to 70 ppb.

Example 2

As listed in Table 1, after 50 g of insulating oil containing about 80 ppm of PCB, 50 g of DMI and 2.5 g of powdery KOH were mixed in a flask, as in Example 1, the mixture was stirred briskly while being maintained at a temperature of 200° C for about 6 hours. After cooling the mixture to room temperature, the lower layer of DMI was removed. Then the PCB in the oil layer was analyzed by a GC-MS, whereby it was confirmed that the PCB content had decreased to below the detection limit value of the GC-MS. Under the conditions of this example, the initial blending ratio of alkalis with respect to the whole reaction system corresponds to 25,000 mg/kg, and the ratio of alkalis with respect to the chlorine amount in the PCB corresponds to 720 in terms of mole ratio. Note that after the treatment was carried out for about 2 hours under the same conditions, it was confirmed that the concentration of the PCB in the oil layer had decreased to 90 ppb.

Example 3

As listed in Table 1, after 50 g of insulating oil containing about 80 ppm of PCB, 100 g of DMI, 1.5 g of powdery NaOH and 1.0 g of powdery CaO were mixed in a flask, as in Example 1, the mixture was stirred briskly while being

maintained at a temperature of 210° C for about 6 hours. Note that CaO also functions as an alkali. After cooling the mixture to room temperature, the lower layer of DMI was removed. Then the PCB in the oil layer was analyzed by a GC-MS, whereby it was confirmed that the PCB content had decreased to below the detection limit value of the GC-MS. Under the conditions of this example, the initial blending ratio of alkalis with respect to the whole reaction system corresponds to 17,000 mg/kg, and the ratio of alkalis with respect to the chlorine amount in the PCB corresponds to 90 in terms of mole ratio. Note that after the treatment was carried out for about 2 hours under the same conditions, it was confirmed that the concentration of the PCB in the oil layer had decreased to 80 ppb.

Example 4

As listed in Table 1, after 50 g of insulating oil containing about 8,000 ppm of PCB, 150 g of DMI and 2.5 g of powdery KOH were mixed in a flask, as in Example 1, the mixture was stirred briskly while being maintained at a temperature of 210 ° C for about 4 hours. After cooling the mixture to room temperature, the lower layer of DMI was removed. Then the PCB in the oil layer was analyzed by a GC-MS, whereby it was confirmed that the PCB content had decreased to below the detection limit value of the GC-MS. Under the conditions of this example, the initial blending ratio of alkalis with respect to the whole reaction system corresponds to 12,500 mg/kg, and the ratio of alkalis with respect to the chlorine amount in the PCB corresponds to 7 in terms of mole ratio. Note that after the treatment was carried out for about 2 hours under the same conditions, it was confirmed that the concentration of the PCB in the oil layer had decreased to 50 ppb.

Example 5

As listed in Table 1, after 50 g of insulating oil containing about 8,000 ppm of PCB, 50 g of DMI, 2.8 g of powdery CaO and 2.0 g of powdery NaOH were mixed in a flask, as in Example 1, the mixture was stirred briskly while being maintained at a temperature of 190° C for about 5 hours. After cooling the mixture to room temperature, the lower layer of DMI was removed. Then the PCB in the oil layer was analyzed by a GC-MS, whereby it was confirmed that the PCB content had decreased to below the detection limit value of the GC-MS. Under the conditions of this example, the initial blending ratio of alkalis with respect to the whole reaction system corresponds to 48,000 mg/kg, and the ratio of alkalis with respect to the chlorine amount in the PCB corresponds to 16 in terms of mole ratio. Note that after the treatment was carried out for about 3 hours under the same conditions, it was confirmed that the concentration of the PCB in the oil layer had decreased to 40 ppb.

Example 6

As listed in Table 1, after 50 g of insulating oil containing about 80 ppm of PCB, 150 g of DMI and 3.8 g of powdery NaOH were mixed in a flask, as in Example 1, the mixture was stirred briskly while being maintained at a temperature of 210° C for about 5 hours. After cooling the mixture to room temperature, the lower layer of DMI was removed. Then the PCB in the oil layer was analyzed by a GC-MS, whereby it was confirmed that the PCB content had decreased to below the detection limit value of the GC-MS. Under the conditions of this example, the initial blending ratio of alkalis with respect to the whole reaction system corresponds to 19,000 mg/kg, and the ratio of alkalis with respect to the chlorine amount in the PCB corresponds to 1500 in terms of mole ratio. Note that after the treatment was carried out for about 2 hours under the same conditions, it was confirmed that the concentration of the PCB in the oil layer had decreased to 30 ppb.

Example 7

As listed in Table 1, after 50 g of insulating oil containing about 8,000 ppm of PCB, 150 g of DMI and 5.5 g of powdery KOH were mixed in a flask, as in Example 1, the mixture was stirred briskly while being maintained at a temperature of 200 ° C for about 3 hours. After cooling the mixture to room temperature, the lower layer of DMI was removed. Then the PCB in the oil layer was analyzed by a GC-MS, whereby it was confirmed that the PCB content had decreased to below the detection limit value of the GC-MS. Under the conditions of this example, the initial blending ratio of alkalis with respect to the whole reaction system corresponds to 27,500 mg/kg, and the ratio of alkalis with respect to the chlorine amount in the PCB corresponds to 16 in terms of mole ratio. Note that after the treatment was carried out for about 2 hours under the same conditions, it was confirmed that the concentration of the PCB in the oil layer had decreased to 20 ppb.

Comparative Example 1

As a first comparative example with respect to Examples 1 to 7, the following investigation was carried out. As listed in Table 1, after 50 g of insulating oil containing about 80 ppm of PCB, 100 g of DMI and 0.3 g of powdery NaOH were mixed in a flask, as in Example 1, the mixture was stirred briskly while being maintained at a temperature of 210° C for about 6 hours. After cooling the mixture to room temperature, the lower layer of DMI was removed. Then the PCB in the oil layer was analyzed by a GC-MS, whereby it was confirmed that 4,000 ppm of PCB had remained. Note that after the treatment was carried out for about 4 hours under the same condition, it was confirmed that 5000 ppb of PCB had remained.

Although the ratio of alkalis with respect to the chlorine amount in the PCB is 120 in terms of mole ratio, which is the same as or higher than that in Examples 1 to 7, a high concentration of PCB remained. The reason for this is that although the amount of NaOH is larger than that of PCB, the initial blending ratio of alkalis with respect to the whole reaction system is 2,000 mg/kg and is much lower than that in Examples 1 to 7.

Example 8

In Examples 1 to 7, 50 g of insulating oil containing PCB was added to the reaction system. In Examples 8 to 10, an extremely small amount of insulating oil containing PCB was added to the reaction system. In these cases, since the quantity of the insulating oil is small, the insulating oil itself dissolves in the DMI layer. Therefore, the concentration of the PCB in the DMI layer was analyzed by a GC-MS after the reaction.

As listed in Table 1, after 3 g of insulating oil containing PCB, 90 g of DMI and 13 g of powdery NaOH were mixed in a flask, as in Example 1, the mixture was stirred briskly while being maintained at a temperature of 200° C for about 5 hours. After cooling the mixture to room temperature, the PCB in the DMI layer was analyzed by a GC-MS. Thereby it was confirmed that the PCB concentration, which had initially been 70,000 ppm with respect to the whole reaction system, had decreased to below the detection limit value of the GC-MS. Under the condition of this example, the initial blending ratio of alkalis corresponds to 130,000 mg/kg, and the ratio of alkalis with respect to the chlorine amount in the PCB corresponds to 3 in terms of mole ratio. Note that after the treatment was carried out for about 2 hours under the same conditions, it was confirmed that the PCB concentration had decreased to 70 ppb.

Example 9

As listed in Table 1, after 3 g of insulating oil containing PCB, 90 g of DMI and 16 g of powdery KOH were mixed in a flask, as in Example 8, the mixture was stirred briskly while being maintained at a temperature of 200° C for about 4 hours. After cooling the mixture to room temperature, the PCB in the DMI layer was analyzed by a GC-MS. Thereby it was confirmed that the PCB concentration, which had initially been 70,000 ppm with respect to the whole reaction system, had decreased to below the detection limit value of the GC-MS. Under the condition of this example, the initial blending ratio of alkalis corresponds to 160,000 mg/kg, and the ratio of alkalis with respect to the chlorine amount in the PCB corresponds to 2.6 in terms of mole ratio. Note that after the treatment was carried out for about 2 hours under the same conditions, it was confirmed that the PCB concentration had decreased to 25 ppb.

Example 10

As listed in Table 1, after 0.3 g of insulating oil containing PCB, 99 g of DMI, 1.5 g of powdery KOH and 1.0 g of powdery CaO were mixed in a flask, as in Example 8, the mixture was stirred briskly while being maintained at a temperature of 210° C for about 5 hours. After cooling the mixture to room temperature, the PCB in the DMI layer was analyzed by a GC-MS. Thereby it was confirmed that the PCB concentration, which had initially been 7,000 ppm with respect to the whole reaction system, had decreased to below the detection limit value of the GC-MS. Under the condition of this example, the initial blending ratio of alkalis corresponds to 25,000 mg/kg, and the ratio of alkalis with respect to the chlorine amount in the PCB corresponds to 4 in terms of mole ratio. Note that after the treatment was carried out for about 2 hours under the same conditions, it was confirmed that the PCB concentration had decreased to 60 ppb.

Example 11

In Examples 1 to 10, insulating oil containing PCB was added to the reaction system. In Examples 11 to 14, on the other hand, instead of PCB being added to the reaction system directly, insulating oil was not added. In these cases too, the concentration of the PCB in the DMI layer was analyzed by a GC-MS.

As listed in Table 1, after 100 g of DMI, 1.9 g of powdery NaOH and PCB of 10,000 ppm of concentration to the whole reaction system were mixed in a flask, as in Example 8, the mixture was stirred briskly while being maintained at a temperature of 200° C for about 5 hours. After cooling the mixture to room temperature, the PCB in the DMI layer was

analyzed by a GC-MS. Thereby it was confirmed that the PCB concentration had decreased to below the detection limit value of the GC-MS. Under the conditions of this example, the initial blending ratio of alkalis corresponds to 19,000 mg/kg, and the ratio of alkalis with respect to the chlorine amount in the PCB corresponds to 3 in terms of mole ratio. Note that after the treatment was carried out for about 2 hours under the same conditions, it was confirmed that the PCB concentration had decreased to 35 ppb.

Example 12

As listed in Table 1, after 100 g of DMI, 2.6 g of powdery KOH and PCB of 10,000 ppm of concentration to the whole reaction system were mixed in a flask, as in Example 8, the mixture was stirred briskly while being maintained at a temperature of 200° C for about 4 hours. After cooling the mixture to room temperature, the PCB in the DMI layer was analyzed by a GC-MS. Thereby it was confirmed that the PCB concentration had decreased to below the detection limit value of the GC-MS. Under the conditions of this example, the initial blending ratio of alkalis corresponds to 26,000 mg/kg, and the ratio of alkalis with respect to the chlorine amount in the PCB corresponds to 3 in terms of mole ratio. Note that after the treatment was carried out for about 2 hours under the same conditions, it was confirmed that the PCB concentration had decreased to 30 ppb.

Example 13

As listed in Table 1, after 100 g of DMI, 1.0 g of powdery NaOH, 1.0 g of powdery CaO and PCB of concentration of 10,000 ppm to the whole reaction system were mixed in a flask, as in Example 8, the mixture was stirred briskly while being maintained at a temperature of 200° C for about 6 hours. After cooling the mixture to room temperature, the PCB in the DMI layer was analyzed by a GC-MS. Thereby it was confirmed that the PCB concentration had decreased to below the detection limit value of the GC-MS. Under the conditions of this example, the initial blending ratio of alkalis corresponds to 20,000 mg/kg, and the ratio of alkalis with respect to the chlorine amount in the PCB corresponds to 2.8 in terms of mole ratio. Note that after the treatment was carried out for about 2 hours under the same conditions, it was confirmed that the PCB concentration had decreased to 40 ppb.

Example 14

As listed in Table 1, after 100 g of DMI, 39 g of powdery KOH and PCB of concentration of 10,000 ppm to the whole reaction system were mixed in a flask, as in Example 8, the mixture was stirred briskly while being maintained at a temperature of 200° C for about 3 hours. After cooling the mixture to room temperature, the PCB in the DMI layer was analyzed by a GC-MS. Thereby it was confirmed that the PCB concentration had decreased to below the detection limit value of the GC-MS. Under the conditions of this example, the initial blending ratio of alkalis corresponds to 390,000 mg/kg, and the ratio of alkalis with respect to the chlorine amount in the PCB corresponds to 4.5 in terms of mole ratio. Note that after the treatment was carried out for about 1 hour under the same conditions, it was confirmed that the PCB concentration had decreased to 100 ppb.

Comparative Example 2

As a second comparative example with respect to Examples 8 to 14, the following investigation was carried out. As listed in Table 1, after 100 g of DMI, 0.4 g of powdery NaOH and PCB of concentration of 10,000 ppm to the whole reaction system were mixed in a flask, as in Example 8, the mixture was stirred briskly while being maintained at a temperature of 200° C for about 6 hours. After cooling the mixture to room temperature, the PCB in the DMI layer was analyzed by a GC-MS. Thereby it was confirmed that 2,000 ppm of PCB had remained. Note that after the treatment was carried out for about 2 hours under the same conditions, it was confirmed that 8,000 ppb of PCB had remained.

The reason why such a high concentration of PCB remained was that the initial blending ratio of alkalis with respect to the whole reaction system is 4,000 mg/kg and is much lower than that in Examples 8 to 14, rather than that the ratio of alkalis with respect to the chlorine amount in the PCB is as low as 0.6 in terms of mole ratio.

Effects of the Examples

As is clear from the above examples, it was confirmed that in Comparative Examples 1 and 2 the PCB concentration had not decreased to 1 ppm or less, while in Examples 1 to 14 PCB had been decomposed to the extent that the PCB concentration might decrease to 1 ppm or less, and further to below the detection limit value.

Other Examples

The conditions under which PCB was decomposed by contacting PCB and alkalis in a non-proton polar solvent, in addition to those in the above examples, were investigated by varying the alkaline amount when the reaction started, the contact temperature of PCB and alkalis, and their contact time. Thereby it was confirmed that PCB could be decomposed with certainty to the extent that the PCB concentration might decrease to below the detection limit by contacting PCB and alkalis at a temperature ranging from about 150 ° C to about 300° C for about 1 to about 10 hours, and by making the blending ratio of alkalis with respect to the whole reaction system when the reaction started 5,000 mg/kg or more. It was further confirmed that by making the blending ratio of alkalis with respect to the whole reaction system when the reaction started 7,000 mg/kg or more, it was possible to decompose PCB with more certainty to the extent that the PCB concentration might decrease to below the detection limit.

Halogenated aromatic compounds other than PCB can be decomposed in the same method, and polychlorinated terphenyl, polybrominated biphenyl and analogous compounds thereof, for instance, can be decomposed to the extent that the content may decrease to below the detection limit.

In the techniques according to the present invention, as alkalis, NaO, Mg(OH)₂ and others may be used as well as NaOH, KOH, CaOH and CaO.

In the above examples, as a non-proton polar solvent, DMI was used, but tetramethylene sulfone or a mixture of DMI and tetramethylene sulfone may be employed as well. It is possible to use a mixture of these solvents and dimethyl sulfoxide, N-methyl pyrrolidone, tetramethyl urea, diethylene glycol or polyethylene glycol dimethyl ether. In this case, considering the alkali resistance at a high temperature, it is preferred that the blending ratio of dimethyl sulfoxide, N-methyl pyrrolidone, tetramethyl urea, diethylene glycol or polyethylene glycol dimethyl ether be 35 % or less.

Table 1-1

Condition	Amount of insulating oil containing PCB (g) In () is PCB concentration in insulating oil (ppm)	Non-proton polar solvent In () is solvent amount (g)	Alkali amount (g) In () is total concentration (mg/kg)	Ratio of alkalis to chlorine (mole ratio)	Processing temperature (°C)	Processing time (hr)	Amount of remaining PCB (ppb)
Inventive examples	1	50 (80)	DMI (100)	NaOH 2.0 (13,000)	800	2	70
	2	50 (80)	DMI (50)	KOH 2.0 (25,000)	720	2	90
	3	50 (800)	DMI (100)	NaOH 1.5 CaO 1.0 (17,000)	90	2	80
	4	50 (8,000)	DMI (150)	KOH 2.5 (12,500)	7	2	50
	5	50 (8,000)	DMI (50)	CaO 2.8 NaOH 2.0 (48,000)	16	3	40
	6	50 (80)	DMI (150)	NaOH 3.8 (19,000)	1500	2	30
	7	50 (8,000)	DMI (150)	KOH 5.5 (27,500)	16	2	20
							N.D.
							N.D.
							N.D.
							N.D.
							N.D.
							N.D.
							N.D.
Comparative example 1							5,000
							4,000

Table 1-2

Condition	Amount of insulating oil containing PCB (g) In () is PCB concentration in whole system (ppm)	Non-proton polar solvent In () is solvent amount (g)	Alkali amount (g) In () is total concentration (mg/kg)	Ratio of alkali to chlorine (mole ratio)	Processing temperature (°C)	Processing time (hr)	Amount of remaining PCB (ppb)
Inventive examples	8	3 (70,000)	DMI (90)	NaOH 13 (130,000)	3	200	70
	9	3 (70,000)	DMI (90)	KOH 16 (160,000)	2.6	200	N.D.
	10	0.3 (7,000)	DMI (99)	KOH 1.5 CaO 1.0 (25,000)	4	210	60
						2	60
						5	N.D.

Table 1-3

Condition	PCB addition concentration to whole system (ppm)	Non-proton polar solvent In () is solvent amount (g)	Alkali amount (g) In () is total concentration (mg/kg)	Ratio of alkalis to chlorine (mole ratio)	Processing temperature (°C)	Processing time (hr)	Amount of remaining PCB (ppb)
Inventive examples	11	10,000	DMI (100)	3	200	2	35
						5	N.D.
	12	10,000	DMI (100)	3	200	2	30
						4	N.D.
13	10,000	DMI (100)	NaOH 1.0 CaO 1.0 (20,000)	2.8	200	2	40
						6	N.D.
14	100,000	DMI (100)	KOH 39 (390,000)	4.5	200	1	100
						3	N.D.
Comparative example 2	10,000	DMI (100)	NaOH 0.4 (4,000)	0.6	200	2	8,000
						6	2,000

Industrial Applicability

As described in the foregoing, in accordance with the present invention, halogenated aromatic compounds and alkalis are contacted in a non-proton polar solvent at a temperature ranging from about 150° C to about 300° C for about 1 to about 10 hours, and the blending ratio of alkalis with respect to the whole reaction system when the reaction starts is set at 5,000 mg/kg or more. Therefore, in accordance with the present invention, it is possible to remove halogenated aromatic compounds such as PCB, which, even in small quantities, are directly hazardous to the human body with certainty and safely, to the extent that such compounds are rendered substantially harmless. Accordingly, it is possible to treat hydrocarbon oil containing PCB and others to the extent that such compounds are rendered substantially harmless.

Claims

1. A method of decomposing halogenated aromatic compounds using alkalis by contacting the halogenated aromatic compounds and the alkalis in a non-proton polar solvent, wherein the halogenated aromatic compounds and the alkalis are contacted at a temperature ranging from about 150° C to about 300° C for about 1 to about 10 hours, and the blending ratio of said alkalis with respect to the whole reaction system when the reaction starts is 5,000 mg/kg or more.
2. The method of decomposing halogenated aromatic compounds using alkalis according to claim 1, in which the blending ratio of said alkalis with respect to the whole reaction system when the reaction starts is 7,000 mg/kg or more.
3. The method of decomposing halogenated aromatic compounds using alkalis according to claim 1, in which said halogenated aromatic compounds are one halogenated aromatic compound selected from a group consisting of polychlorinated biphenyl, polychlorinated terphenyl, polybrominated biphenyl, and analogous compounds thereof, or a mixture of two or more halogenated aromatic compounds selected from said group.
4. The method of decomposing halogenated aromatic compounds using alkalis according to claim 1, in which said halogenated aromatic compounds are added to said non-proton polar solvent after being diluted by hydrocarbon oil, the principal component of which is non-aromatic hydrocarbon, to the extent that the concentration of the compounds ranges from 2 ppm to 80 %.
5. The method of decomposing halogenated aromatic compounds using alkalis according to any one of claims 1 to 4, in which said alkalis are one alkali selected from a group consisting of sodium hydroxide, potassium hydroxide, calcium hydroxide and magnesium hydroxide, or a mixture of two or more alkalis selected from said group.
6. The method of decomposing halogenated aromatic compounds using alkalis according to any one of claims 1 to 4, in which said non-proton polar solvent is a solvent selected from a group consisting of 1,3-dimethyl-2-imidazolidinone, tetramethylene sulfone, and a mixture of 1,3-dimethyl-2-imidazolidinone and tetramethylene sulfone.
7. The method of decomposing halogenated aromatic compounds using alkalis according to any one of claims 1 to 4, in which said non-proton polar solvent is a mixture the principal component of which is a solvent selected from a group consisting of 1,3-dimethyl-2-imidazolidinone, tetramethylene sulfone, and a mixture of 1,3-dimethyl-2-imidazolidinone and tetramethylene sulfone, and which contains one polar solvent selected from dimethyl sulfoxide, N-methyl pyrrolidone, tetramethyl urea, diethylene glycol and polyethylene glycol dimethyl ether, or two or more polar solvents selected from said group at a concentration of 35 % or less.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP95/00785

A. CLASSIFICATION OF SUBJECT MATTER		
Int. Cl ⁶ A62D3/00		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED		
Minimum documentation searched (classification system followed by classification symbols)		
Int. Cl ⁶ A62D3/00		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Jitsuyo Shinan Koho 1926 - 1995		
Kokai Jitsuyo Shinan Koho 1971 - 1995		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP, 49-126651, A (Kanegafuchi Chemical Industry Co., Ltd.), December 4, 1974 (04. 12. 74), Page 2 (Family: none)	1 - 7
<input type="checkbox"/> Further documents are listed in the continuation of Box C. <input type="checkbox"/> See patent family annex.		
* Special categories of cited documents: "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier document but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family		
Date of the actual completion of the international search		Date of mailing of the international search report
July 18, 1995 (18. 07. 95)		August 1, 1995 (01. 08. 95)
Name and mailing address of the ISA/ Japanese Patent Office		Authorized officer
Facsimile No.		Telephone No.

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