

# PATENT SPECIFICATION

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## (54) ADDUCTS OF ORGANOMETALLIC COMPOUNDS WITH THIOUREA

(71) We, INSTITUT ELEMENTO-ORGANICHESKIKH SOEDINENY AKADEMII NAUK SSSR, of ulitsa Vavilova 28, Moscow, USSR., do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed to be particularly described in and by the following statement:—

This invention is concerned with the separation of certain organometallic compounds from mixtures containing them, and with novel adducts formed during this separation and with a method of making the adducts.

Organometallic compounds of the formula:



wherein L is an unsubstituted cyclopentadienyl or benzene ring; I' is an unsubstituted cyclopentadienyl or benzene ring, or represents an appropriate number of carbonyl groups; and M is a transition metal; are known. In the preparation of these compounds, it is necessary to separate them from the preparative reaction mixture. We have now found that these organometallic compounds form adducts with thiourea, whereas the other components of the reaction mixture generally do not, and that the compounds can conveniently be separated from the mixture by forming an adduct with thiourea, the adduct being thereafter decomposed to liberate the free organometallic compound. The adducts themselves are novel and form one aspect of the present invention.

The invention provides an adduct of thiourea with an organometallic compound of the formula:



wherein L is an un-substituted cyclopentadienyl or benzene ring; L' is an unsubstituted cyclopentadienyl or benzene ring or represents an appropriate number of carbonyl groups; and M is a transition metal.

The invention also provides a method of making such an adduct, which comprises treating the organometallic compound with thiourea in the presence of an organic solvent which comprises an alkanol having from 1 to 3 carbon atoms or a dialkylketone, or a mixture of two or more thereof.

The invention further includes a method of separating an organometallic compound of the formula:



where L, M and L' are as defined above, from a mixture thereof with other substances which do not form an adduct with thiourea, which comprises treating the mixture with thiourea in the presence of an organic solvent; separating the thiourea adducts so formed; and recovering the organometallic compound from the adducts.

In the methods of the invention, the organometallic compound (which expression is used hereinafter to refer to the compounds LML' and not to the adducts) is treated with thiourea in a medium which comprises an alkanol having from 1 to 3 carbon atoms, or a dialkylketone, or a mixture of two or more thereof. The said medium may also contain benzene, an alkyl benzene, carbon tetrachloride or petroleum ether.

Alkanols may be used both as the reaction medium and as initiators of the reaction between the organometallic compounds and thiourea.

When an alkanol having from 1 to 3 carbon atoms is used primarily as initiator and not solvent, it should be used in a quantity of not more than 20 percent by weight of the thiourea, and the treatment with thiourea should be carried out in a medium of benzene or an alkyl derivative thereof.

When the treatment with thiourea is carried out using, as solvent, an alkanol having from 1 to 3 carbon atoms, a dialkylketone, or a mixture of said alkanol or dialkylketone with benzene, an alkyl derivative thereof, carbon tetrachloride or petroleum ether, in which mixture the amount of alkanol



Elizabeth the Second by the Grace of God of the United Kingdom of Great Britain and Northern Ireland and of Her other Realms and Territories, Queen, Head of the Commonwealth, Defender of the Faith: To all to whom these presents shall come greeting:

WHEREAS a request for the grant of a patent has been made by

Institut Elementoorganicheskikh Soedineny Akademii Nauk SSSR, of ulitsa Vavilova 28, Moscow, Union of Soviet Socialist Republics, a Russian body corporate,

for the sole use and advantage of an invention for

Adducts of organometallic compounds with thiourea:

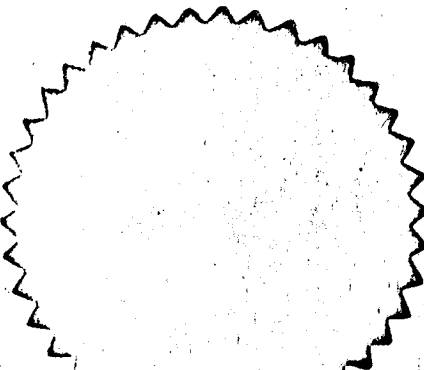
AND WHEREAS We, being willing to encourage all inventions which may be for the public good, are graciously pleased to condescend to the request:

KNOW YE, THEREFORE, that We, of our especial grace, certain knowledge, and mere motion do by these presents, for Us, our heirs and successors, give and grant unto the person(s) above named and any successor(s), executor(s), administrator(s) and assign(s) (each and any of whom are hereinafter referred to as the patentee) our especial licence, full power, sole privilege, and authority, that the patentee or any agent or licensee of the patentees and no others, may subject to the conditions and provisions prescribed by any statute or order for the time being in force at all times hereafter during the term of years herein mentioned, make, use, exercise and vend the said invention within our United Kingdom of Great Britain and Northern Ireland, and the Isle of Man, and that the patentee shall have and enjoy the whole profit and advantage from time to time accruing by reason of the said invention during the term of sixteen years from the date hereunder written of these presents: AND to the end that the patentee may have and enjoy the sole use and exercise and the full benefit of the said invention, We do by these presents for Us, our heirs and successors, strictly command all our subjects whatsoever within our United Kingdom of Great Britain and Northern Ireland, and the Isle of Man, that they do not at any time during the continuance of the said term either directly or indirectly make use of or put in practice the said invention, nor in anywise imitate the same, without the written consent, licence or agreement of the patentee, on pain of incurring such penalties as may be justly inflicted on such offenders for their contempt of this our Royal Command, and of being answerable to this patentee according to law for damages thereby occasioned:

PROVIDED ALWAYS that these letters patent shall be revocable on any of the grounds from time to time by law prescribed as grounds for revoking letters patent granted by Us, and the same may be revoked and made void accordingly:

PROVIDED ALSO, that nothing herein contained shall prevent the granting of licences in such manner and for such considerations as they may by law be granted: AND lastly, We do by these presents for Us, our heirs and successors, grant unto the patentee that these our letters patent shall be construed in the most beneficial sense for the advantage of the patentee.

IN WITNESS whereof We have caused these our letters to be made patent as of the twenty-third day of August one thousand nine hundred and seventy-four and to be sealed.



*Edward Aming*