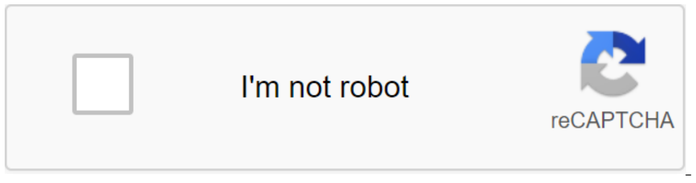

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Best solvent for recrystallization of anthracene

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Thanks to multiple washes of raw anthracene with xylein, DMF-19.96% of isopropanolamine and chlorobenzen, it was possible to get anthracene and carbazole purity above 98 w%. © 2013 AIChE J American Institute of Chemical Engineers, 60: 275-281, 2014 Ye Gao, Teng Chang, Changyou Yu, Peiyi Li, Ningning Tian, Songgu Wu, Effect of solvents on solid-liquid phase equilibrium 1.3-Di-o-tolguanidine, Journal of Molecular Fluids, 10.1016/j.molliq.2020.113147, (113147), (2020). Ji-Hao Ma, Xian Yong Wei, Min-Yao zhou, Guang-Hui Liu, Fan-Jing Liu, Joon Tsyu Liu, Xin-yue Yu, Ji-Ming Tsong, insulation and cleaning of carbazolo contained in anthracian slag by extraction in combination with liquid medium pressure chromatography, Chinese Journal of Chemical Engineering, 10.1016/j.cjche.2018.012, (2019). 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KLEISS ETAL 2,767,232 SEPARATION AND PURIFICATION OF ANTRACEEN BY THE CRY OF STALLIZATION FROM THE DIOXAN SOLUTION FILED JUNE 30. 1952 I60 I80 TEMPERATURE,F. 60 SOLUBILITY CURVES, CRUDE ANTHRACENE IN VARIOUS ORGANIC SOLVENTS F 2 DJ 3 o (I) D N: O o o (I) A FURIFURAL EL DIOXAN IO' IN V EN TORS D. KLEISS A. D. ADAMS A TTORNE United States Patent 1 fiice 7 2,767,232 Patented October 16, 19 56 SEPARATION AND PCATION OF ANTHRA- CENE BY CRYSTALLIZATION FROM A DIOX- ANE SOLUTION Louis D. Kleiss and Archie Archie 1). Adams, Berger, ten, as a signatory to the Phillips Oil Company, Delaware Corporation applications June 30, 1952, Serial No. 296,467 Claims. This invention refers to the separation and purification of crystallized resin derivatives. In one of its aspects, this invention involves separating and cleaning the anthrazen from anthrazene oil when distilling coal resin. In another of its aspects, this invention refers to the improvement of the solvent for the crystallization of the anthrazcan in the presence of carbasol, phenantrene and other components contained in anthrazen oil in the distillation of coal resin. Antracen is an aromatic organic compound with the CHHID empirical formula. It is found in very small amounts, about one percent, in coal resin. It is also found in the remnants of distillation of cracked petroleum products. Antracen as a chemical has many uses, but its most important use as a starting point in the creation of alisarine, alysari dye stufis, and aniline coloring nutrition. It is also used as a reagent in the adoption of emulsified and fungicide drugs. It is also an ingredient in varnishes, paints, varnishes, doping and enamel containing various simple cellulose esters such as cellulose acetate and nitrocellulose, or various mixed cellulose esters such as cellulose acetopropionate (added to enhance the life of the film by absorbing ultraviolet rays). Antracen is also an ingredient in printing ink used for printing on banknotes, checks and other fiscal papers, secret numbers and marks that are visible under the influence of ultraviolet rays of light and rays Lamp. As mentioned, an important important anthrace is a charcoal resin. Raw charcoal resin is distilled and anthrazen oil cut, boiling in the range of 572 to 662 F. going. This incision, which is about nine percent of the original crude coal resin, contains fuoren, phenantren, anthrazen, carbazole and other unidentified components. Antrazen is present in this faction in the amount of 10 to percent. When the anthracazen oil is cut, the distillate crystallizes to form a raw cake that can be separated from the oily component of centrifuge, filtration or other means. The composition of the cake, which is called raw anthracazen, depends on etficiency and method of operation so far, and the concentration of anthrazene in said cakes ranged from about 5 to 35 percent. The usual procedure of further concentration of anthracene involves recrystalization of selective solvent. The most common solvents used for this purpose are furfiural and pyridine, as well as homologies of each of them. The nafta solvent is also widely used. In order to get antracenus from 80 to 90 percent purity, which is the minimum necessary for the further use of anthracene, it is necessary to use a number of stages of crystallization from solvents. We have now discovered an excellent solvent from which to crystallize the anthrax, consisting of dioxane, also known as dioxane, 1.4-dioxane, 1.4-dithylene oxide, dioxiethylene ether, or dioxythilen. The main drawback of the use of fur solvent as such for the crystallization of anthrazen is its tendency to form a black polymer, especially at high temperatures. This means that fur should be used at low temperatures not higher than 140 F to make the polymer form a blacker anthrace product and makes it unsuitable for dye production. At such low temperatures per unit of harvest will be used large volumes of furfal (4-6 times more than in the case of dioxane, where only the boiling point of the solvent is the limit), and the yield will be low if the anthrac is not sucked out in refrigeration conditions. In addition, any fur mother liqueur sticking to the antracen product should be removed by rinsing with another solvent or high vacuum drying to avoid high temperatures. In addition, the fur in the vault becomes blackened and must be cleaned before use. The drawbacks of the pyridine (and its homologers) include a cost that is twice as much as dioxan'e; it is tertiary amin, exhibiting chemical reactivity common to amines. This leads to a high loss of solvent. In addition, the smell of pyridine is shrill and undesirable, and it is difiicult to handle. It is the object of this invention to provide an improved process for separating and cleaning crystallized Resin. Another purpose of this invention is to ensure an improved process of separation and anthrazena from raw anthracaene, which includes phenantren, carbazole and fluoride as impurities. Another object of this invention is the provision of an improved solvent for the crystallization of the anthrax in the presence of phenantren, carbasol and other impurities. Other objects and advantages of this invention will be obvious to those who are skilled in art from concomitant disclosure and discussion. The accompanying picture is a graphic presentation and data comparison. Our invention is in the discovery that a solvent consisting of dioxane, also known as dioxane, 1.4-dioxane, 1.4-dithylene oxide, dioxethylene ether, or dioxide dioxide diethyl (O-GH -CH OCHzHr) is an excellent solvent from which crystallized anthrax mixed with impurities such as fenantren, carb and fluoride. weak, pleasant smell, completely wrong with water and with most organic solvents. It is neutral, stable to the heat and stable in relation to most chemicals. it has been reported that 1.4-dioxane tends to form explosive peroxide when allowed to stand, and they should be neutralized with the appropriate inhibitor before distilling the attempt. Since dioxane is heat stable it can be restored in compact, inexpensive solvent recovery systems. It's also moderate in cost. While 1.4-dioxane has been proposed as a solvent for a wide range of materials, including vegetable and mineral oils, waxes, fats, lignin, natural resins such as dewaxed dammar, elemi, guaiae, resin and shells, synthetic resins such as ethereal gum, kumar resin, alquid, some types of vinyl resins, and pulp esters and esters, not found in the previous art of use 1.4- This invention includes In general, the dissolution of raw en - therefrom anthrazen crystals improved purity and restoration said crystals from the mother of the liqueur. More specifically, we found that if raw anthrazen. containing at least 25 percent weight of anthrazen is dis-. Resolved in 1.4-dioxane and as a result the solution cooled by 1.4-dioxane is a colorless liquid with however, it has ii 7 Melting R'ange: anthrazen crystals of purity in the region of 80 percent can be restored in one step crystalization. Antracene can be even more pure with more crystallization. While 'th'e present invention can be applied to others I cleanse,' it will be described in 'a few specific ex enough, especially in connection with the cleaning of raw anthracane. Thefollowing examples are not intended to limit our invention unnecessarily, but to assist in i 7 describingit and to assist those who are skilled in the arts in practice. The advantages of our solvent over conventional solvent in the purification of the anthracene. . Raw anthracene used in the following characteristics: Appearance: Similar brown sugar. Oily, paper stains on contact. 270-330 (clean anthracen

melting point, 422 F.; carbazole, 473 F.; phenantren, 209 F.) v v Sublimation: O. 24% sublime at 250 F; and 0.1-2 mm Hg. This is about 565 F. corrected to 760 mm. (Boiling point of pure anthracene 669 F.) Spectroanalysis: Wt. Percentage of anthracene 127.9 carbazole 10.9 phenantren 23.8 Sample solvent containing 36.3 grams of raw anthrax per 100 ml. 1.4-dioxane solvent at 178 F. cooled over a period of time to 60 F. with crystalline sediment formation. The crystals were pressed to remove as much'liquor as possible, and then the dioxane solvent was removed by vacuum flashing at 220 F. Precipitation yields on a washed and dried base amounted to 25.2 percent. The precipitation contained 79.1 per cent of the anthrazen, 2.5 per cent of carbasol and 3.7 per cent of phenantren, representing 71.5 per cent of the anthrazen crop. Example 2 Fresh distilled fur has been used to avoid depositing the polymer on the sediment and discoloring it. Solution containing 22.5 grams of raw anthrazen per 100 ml of fur solvent at 180. (which is too hot' for. The resulting sediment was separated from the mother's alcohol filtration. and 'wash with NazSzOs' solution' and then rinsed with water and vacuum dried at 230 F. To remove furfural. , precipitation yields on a washed and dried basis ob- V tained was 29.5 percent. containing 12.4 grams of raw anthracene per 100 ml of Stoddard solvent at 230 F. was cooled to 75 F. The resulting crystalline sediment was separated by dotted lines indicating the behavior of partially dissolved raw i Total raw anthrax. 7 present is shown by the order of crossing a hard and dotted line. At the temperature indicated by the junction abscisses, all this raw anthracene is soluble; As the temperature drops, the less soluble components will crystallize; To find the quantity still in the solution, follow'theidotted line the'intersection temperature ofjco'olin'g; The order at this point will re-resent the grams of material stillin' decision; lGrammes are precipitated cleaned anthrazen can be. received on the difference. Let's say 36.5 grams of raw anthrax in 100 ml of dioxane solvent. Teh. a heavy hard line for dioxane shows that this is the amount of raw anthrax. will completely dissolve in l78-F. As a result of this decision up to 60 F. (follow the dotted line on the left) it is established that 27.2 grams will remain in the solution; so, 9.3 grams of cleaner anthraconenair is deposited. Those who are skilled in art will easily appreciate that the difa and purification of the anthracene, which includes, the dissolution of raw antracene 'in a solvent consisting of dioxane, cooling theresultant crystal crystal solution crystals therefrom'anthracene improved purity of recovery said crystals from the mother of the liquor, redissolving said crystals in 'further amount said solvent, cooling the second result of the decision to 'crystalize therefrom anthracene crystals even greater I. . antracene, which includes the dissolution of crude'anthracene V 3. The process for separating and cleaning the ma solvent consisting essentially - of dioxary, cooling as a result of s0luti0n crystallize therefrom anthrazene recovery said crystals 1 crystals improved purity, and from the mother of the liquor. 7 - 4. The process of separating and cleaning the anthrax, which involves dissolving raw anthrazen containing at least twenty-five percent of the weight of anthra'eene in a solvent consisting essentially ofofdioxane, cooling as a result of a crystallization solution from the anthrax crystals about 80 weight percent purity and recovery said crystals. mother of liquor. 5. YA the process of separating and cleaning the anthracene, which involves dissolving raw anthracene derived from resin and containing at least 25 weights of interest of anthracene in a solvent consisting essentially of dioxane at a temperature of around 175 from the mother's liqueur by filtering and washed with pentan to remove the occlud solvent Stoddard. The precipitation was dried by the oven at 210 F. The yield of precipitation obtained on a washed and dried basis was 33.6%. This precipitation contained 59.6 per cent of the anthrazen, 23.0 per cent of carbasol and 2.5 per cent of phenantren, representing 71.7 per cent of the anthrazen crop. 7 Thus, from the above example we can see that .anthracene about 80 percent purity can be obtained in one crystallization step, using dioxane as a solvent for raw anthrazen. Referring to the pattern, heavy hard lines show grams of .of raw anthracene used that will completely dissolve the inlOO ml solvent at this temperature. 7,350 F., cooling as a result of the decision to crystallize it from the crystals of anthrazen about the weight percentage of purity, and the recovery said crystals from the mother of the liqueur. 1 Links in the olf'this patent UNITED STATESPATENTS. 1,693,713 Jaeger Dec.' 4, 1928. June 7,764,031 .17, 1930 1,879,686 Jaeger et a1. September 27, 1932 2,138,832 Brown et al. December; 6, 1938 OTHER HELP 7 v r 7 Price; J; Am. Chem. 500., vol. 573, page 2101 (1936). 2101*(1936).