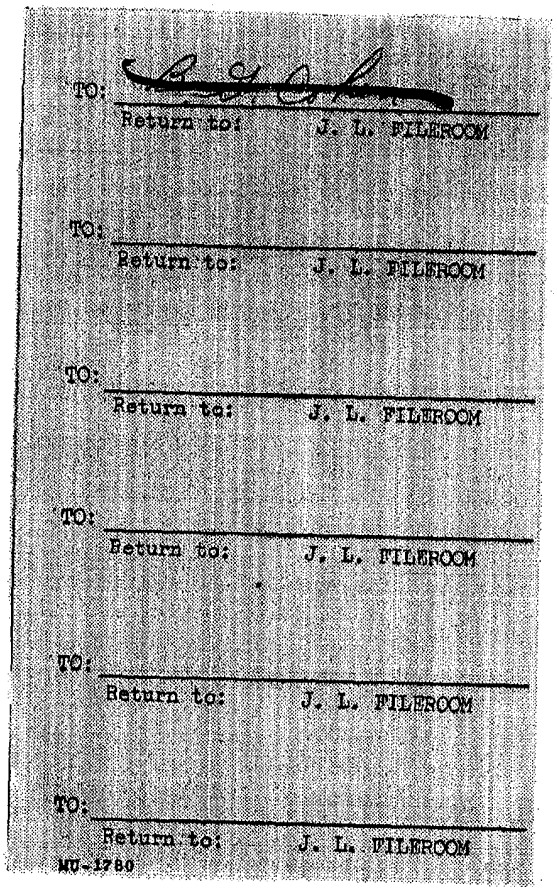
N37230



TO: [redacted]
Return to: J. L. FILEROOM

TO: _____
Return to: J. L. FILEROOM

TO: _____
Return to: J. L. FILEROOM

TO: _____
Return to: J. L. FILEROOM

TO: _____
Return to: J. L. FILEROOM

TO: _____
Return to: J. L. FILEROOM

MU-1780

JLR-33-45, No. 2.

Serial Number 18807.

(1)

Dr. H. W. Elley (2)
Dr. E. K. Bolton (1)
Dr. Wm. Kirk (1)
Mr. A. J. Wuertz
for I.C.I. (3)
J. L. Files (2)
Dr. S. S. Rossander (1)
Mr. H. R. Waldron (1)

E. I. DuPont DeNemours & Company

Jackson Laboratory

June 16, 1944.

2:2'-Dibenzanthronyl - Analytical Method

Problem Report

E. T. Howell

H. R. Lee,
Group Leader

S. S. Rossander,
Division Head

2:2'-Dibenzanthronyl - Analytical Method
(Project No. 2319)

E. T. Howell

JLR-33-45, No. 2. Serial Number 18807

Object of the Investigation:

To develop a reliable method for determining the absolute purity of crude 2:2'-dibenzanthronyl and the percentage of its principal impurities.

Period Covered by the Report:

December 10, 1943 to February 16, 1944.

Historical Background:

2:2'-Dibenzanthronyl is used as an intermediate in the manufacture of Ponsol Jade Green, Ponsol Jade Green Supra, Ponsol Navy Blue, Ponsol Blue Green Y, and its use is proposed in the manufacture of dibenzanthrone, which in turn is used in the manufacture of Ponsol Dark Blue BR, Ponsol Navy Blue RA, Ponsol Dark Blue 2G, and Ponsol Black BN.

2:2'-Dibenzanthronyl has been produced in this country since 1932 only by the DuPont Company under license from General Aniline. U.S. 1,564,423 (Luttringhaus to Badische, 1925), the patent that covered this important intermediate, expired in 1942. It became desirable to improve our position in respect to the manufacture of this product and if possible to obtain patent protection on the improvements. This work is described in JLR-33-45, No. 1, Serial Number 18165 and JLR-33-45, No. 3, Serial Number 18808. In order to prosecute this work more intelligently, it was necessary to have an accurate method for assaying the results. The former method consisted merely of extracting the crude sample with mono-chlorbenzene. The weight of the residue was considered as the weight of pure dibenzanthronyl in the sample. This method is subject to considerable error due to the fact that dibenzanthrone, isodibenzanthrone and ash are not removed, and that the main body has considerable solubility in organic solvents.

I.C.I. Report H-3520, September 1939, described a combined gravimetric and optical method for the analysis of 2:2'-dibenzanthronyl. It gives the percentages of the following components: 2:2'-dibenzanthronyl, benzanthrone, ash, and a fourth fraction containing "Violanthrone B", dibenzanthrone, isodibenzanthrone lumped together.

The present report describes a method based on chromatography and spectrophotometry, which is believed to be more rapid than others described, considering its accuracy.

Conclusions:

The object was attained. A reliable method was developed employing chromatography and spectrophotometry in which 2:2'-dibenzanthronyl and benzanthrone are determined to an accuracy of less than 1% compared with samples of known composition. The hydroxy bodies, dibenzanthrone and isodibenzanthrone, lumped together, are determinedby difference; 100% - (% dibenzanthronyl + % benzanthrone + % ash).

Summary:

As a chromatographic adsorption medium, basic copper carbonate was employed to give sharp separations of the benzanthrone and 2:2'-dibenzanthronyl on the one hand, from hydroxy bodies, dibenzanthrone and iso-dibenzanthrone which stay adsorbed in the chromatograph tube. The impurities in the order of the magnitude of their occurrance are:

1 - Hydroxy bodies, including the "Violanthrone B" of I.C.I., which is possibly a benzanthrone-pinacol formed by the reducing action of methanolic-KOH on benzanthrone. It appears as a violet strata in the chromatograph tube (when using alumina). It adsorbs much less strongly than dibenzanthrone. Occurrance may be as high as 20%.

2 - Dibenzanthrone. Is adsorbed strongly as a narrow dark blue strata at the very top of the tube. It may possibly occur to the extent of 5%.

3 - Benzanthrone. Is not adsorbed. It has been detected from less than 1% to as high as nearly 3%.

4 - Iso-dibenzanthrone. Is adsorbed strongly with the dibenzanthrone layer. This apparently occurs in traces only.

The best combination of adsorbent and solvent was basic copper carbonate with toluene.

Samples of pure 2:2'-dibenzanthronyl and of pure benzanthrone were prepared and the optical properties in mono-chloro-benzene, trimethyl-benzene, and toluene were determined.

Equations were established for the quantitative simultaneous determination of 2:2'-dibenzanthronyl and benzanthrone in the toluene effluent from the chromatograph tube.

Patent Situation:

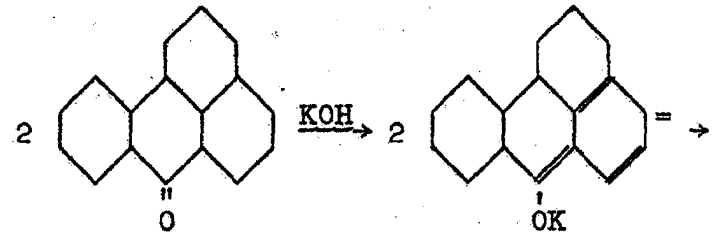
Patent Report AN-2336 failed to reveal the purification of 2:2'-dibenzanthronyl by adsorption with anything except activated carbon. The use of inorganic compounds for this purpose is apparently new.

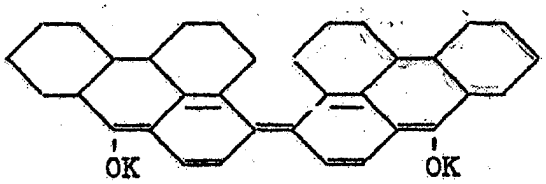
Plans for Future Work:

No further work is planned in regard to analytical methods. The method shown in the Appendix is recommended for use.

Theoretical Discussion:

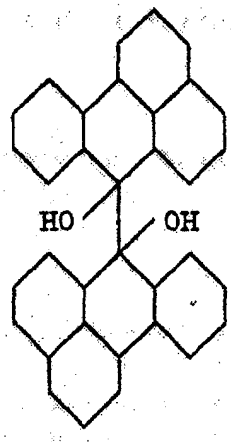
2:2'-Dibenzanthronyl is formed by the methanolic-caustic potash fusion of benzanthrone under conditions milder than that required to form dibenzanthrone. The exact mechanism of this reaction is not known. However, observation has shown that shortly after benzanthrone is introduced into the melt there is a distinct color change. If the material is isolated at this stage by diluting into excess of water, benzanthrone is recovered in substantially unchanged condition. But without isolation, and with further heating, 2:2'-dibenzanthronyl forms more or less rapidly depending upon the temperature and other conditions. Based on these observations it has been postulated that the benzanthrone first undergoes an ionization or enolization, and that two molecules of this transitory form then unite to form a molecule of dibenzanthronyl, according to the following reactions:





In addition to the above main reaction, which at the most goes not more than about 85%, small percentages of dibenzanthrone and isodibenzanthrone are formed by ring-closure of 2:2'-dibenzanthronyl, and by formation and ring-closure of 2-Bz-1'-dibenzanthronyl. In the presence of alcoholic potash it has been suggested that part of the benzanthrone is reduced to a pinacol-like body (I.G. Plant Process of August 1932) analogous to pinacol formation by the reduc-

tion of aliphatic ketones. If the analogy is correct, the formula of this compound would be:



In support of this theory it is known that hydroxy groups are present in at least one of these impurities, since they can be methylated. (Luttringhaus and Neresheimer; Ann. 1929 473 273).

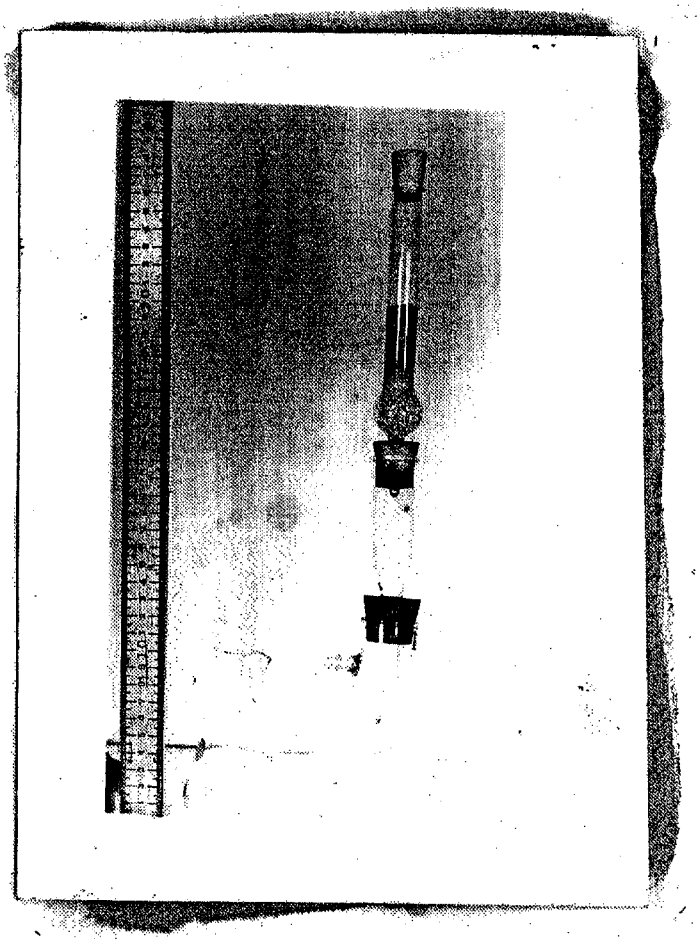
Experimental Results:

The successful quantitative analysis of Bz-1:Bz-1'-dibenzanthronyl by means of combined chromatographic and optical methods (JLR-33-50, No. 1, Serial Number 18733) suggested that a similar method might be applied to the analysis of 2:2'-dibenzanthronyl. It was soon found, however, that the combination of adsorbent and solvent which was best suited to the former method was inoperable in the case of the isomeric 2:2'-dibenzanthronyl.

The product used for working out suitable chromatographic conditions was a composite of six plant batches of 2:2'-dibenzanthronyl crude (batches 95, part 1; 95, part 2; 96, part 1; 97, part 1; 98, part 2; and 99, part 1). The method used for testing for proper conditions was as follows. 10 Milligrams of sample (ground to 100 mesh) were heated to the boil in 75 cc. of solvent in a Howell flask (Figure 1-A) and the solution, which was bright red-violet was allowed to cool and was run through a chromatograph tube made up as shown in Figure 1-B, the height of the column of adsorbent being about 50 mm. Careful observations were made of the following points:

1 - How well were the dark bands of impurities kept in the tube? Did they run down and out when the wash was run through?

2 - Was the bright yellow band of 2:2'-dibenzanthronyl retained in the tube, or how easily was it washed out, leaving the impurities sharply adsorbed?



A B

Figure 1

A summary of the results obtained with various adsorbents and solvents is given in Table 1.

Table 1

No.	Adsorbent	Solvent	Result: Adsorbed	Result: Washed through
1	$Al(OH)_3$ heated at 200°C.	Chloro-benzene	Dibenzanthrone	Oxy bodies, dibenzan-throne & benzanthrone
2	$Al(OH)_3$ heated at 300°C.	Chloro-benzene	Dibenzanthrone, oxy, dibenzan-thronyl	Benzanthrone
3	$Al(OH)_3$ heated at 300°C.	Dichloro-benzene	Dibenzanthrone, oxy, dibenzan-thronyl	Benzanthrone
4	$Al(OH)_3$ heated at 300°C.	Nitro-benzene	Dibenzanthrone, oxy	Dibenzanthronyl, benzanthrone
5	$Al(OH)_3$ heated at 300°C.	Trimethyl-benzene*	Dibenzanthrone, oxy	Dibenzanthronyl, benzanthrone
6	$Al(OH)_3$ heated at 300°C.	Dimethyl-benzene**	Dibenzanthrone, oxy	Dibenzanthronyl par-tially only benzan-throne
7	$Al(OH)_3$ heated at 300°C.	Toluene	Dibenzanthrone, oxy, dibenzan-thronyl partial	Benzanthrone
8	$Al(OH)_3$ heated at 300°C.	Diethyl-benzene (Dow)	Dibenzanthrone	Dibenzanthronyl, oxy partial, benzanthrone
9	$Al(OH)_3$ heated at 300°C.	Isopropyl-benzene (Dow)	Dibenzanthrone, oxy, dibenzan-thronyl	Benzanthrone
10	$Al(OH)_3$ heated at 300°C.	Triethyl-benzene (Dow)	Dibenzanthrone, oxy	Dibenzanthronyl, par-tial; benzanthrone
11	$CuCO_3$, $Cu(OH)_2$ Mallinckrodt	Trimethyl benzene*	Dibenzanthrone, oxy	Dibenzanthronyl, benzanthrone (Note 1)
12	$CuCO_3$, $Cu(OH)_2$ Mallinckrodt	Toluene	Dibenzanthrone, oxy	Dibenzanthronyl, benzanthrone
13	$CuCO_3$, $Cu(OH)_2$ Baker	Trimethyl-benzene*	Dibenzanthrone, oxy	Dibenzanthronyl, benzanthrone (Note 2)
14	$CuCO_3$, $Cu(OH)_2$ Baker	Toluene	Dibenzanthrone, oxy	Dibenzanthronyl, benzanthrone (Note 2)

JLNB-4264, page 135; JLNB-4308, pages 23, 24, 27.

*Barrett "Hi-Flash Naphtha", a mixture of isomeric trimethyl-benzene.
**Commercial mixed xylenes.

Note 1 - Ran through very slow.
Note 2 - Ran through O.K.

Apparently the only combinations suitable were No. 4 (alumina and nitrobenzene), No. 5 (alumina and trimethyl-benzene), 11, 12, 13, and 14 (basic copper carbonate with trimethyl-benzene and toluene. If these methods were to be successful, the effluents must be amenable to simultaneous quantitative analysis for dibenzanthronyl and benzanthrone by means of the spectrophotometer. Nitrobenzene and trimethyl-benzene failed on this score, leaving combination No. 14 as the only alternative. The work on the optical part of the analysis is described below.

Preparation of Pure Standards

For quantitative determinations by means of the spectrophotometer it is necessary to know the optical densities of solutions of the pure compounds at significant regions in the spectrum. This requires the preparation of samples of benzanthrone and 2:2'-dibenzanthronyl of "absolute" purity to be used as optical standards.

Preparation of Pure Benzanthrone

In the manufacture of purified benzanthrone, occasional batches are obtained which have a higher purity than the average. Charge 341, part 2, was selected having the following properties:

Benzene insoluble - .06%
Anthraquinone - .5%

50 Grams of this material was dissolved in 500 cc. boiling solvent naphtha with the addition of 5 grams of Nuchar. The solution was filtered hot through paper. The filtrate was cooled and the resulting crystals were filtered off, washed with small portions of solvent naphtha, then with alcohol, and dried.

Yield dry crystals - 39.6 grams (79.2%)
M.P. Literature - 170°
Found - 173-174°
JLNB-4214, page 97.

Preparation of Pure 2:2'-Dibenzanthronyl

Purified 2:2'-dibenzanthronyl was prepared according to the method described in JLR-33-45, No. 1, Serial Number 18165, Appendix 1,

which consists of slurrying leuco-2:2'-dibenzanthronyl (from the fusion mass) with aqueous pyridine bases, giving bright yellow crystals. These gave a pale yellow solution with no trace of red fluorescence in the usual organic solvents. This is a criterion of purity (Luttringhaus and Neresheimer; Ann. 1929 473, 272). 10 Grams of these crystals were dissolved in 200 grams of nitrobenzene at 185° with the addition of 2 grams of Nuchar. The solution was filtered hot through paper and the filtrate was allowed to cool to room temperature. The resulting bright yellow crystals were filtered off, washed with a few portions of nitrobenzene, then with alcohol, and were dried.

Yield of crystals - 9 grams (90%)
M.P. Literature - 325°
Found - 327.5°
JLNB-4264, page 91

Optical Properties:

Solutions of pure benzanthrone and pure 2:2'-dibenzanthronyl were made up in the solvents which worked well in the chromatograph separations, i.e., nitrobenzene, trimethyl-benzene, and toluene. These were made up as follows. About 10 milligrams of sample was weighed to .0001 gram and dissolved in 100 cc. of solvent in a tared volumetric flask. The flask and contents were weighed and the net weight of the solution obtained. Approximately 10 cc. of this solution were transferred to a second tared 100 cc. volumetric flask, weighed, and made up to 100 cc. with the solvent. From this data the concentration of the dilute solution could be calculated and was on the order of .01 milligram per cc. The optical densities of these solutions were measured with the spectrophotometer, using some of the plain solvent in the compensating cell to compensate for the density of the solvent and cell. The readings were then corrected to standard conditions of .01 mg., 1 cm. cell. The density of the nitrobenzene solutions was so great in the blue end of the spectrum due to the yellowness of the solvent itself, that the machine became sluggish and would not operate properly. In the case of trimethyl-benzene, this trouble was not encountered, but duplicate determinations never checked well. The reason for this was obscure, but it was believed to have something to do with the lack of purity of the solvent which was a commercial mixture of isomeric trimethyl-benzene called "Hi-Flash Naphtha". It is difficult to obtain in completely colorless form, requiring treatment with activated charcoal. Material which appeared colorless had a tendency to become slightly yellow on standing. If pure trimethyl-benzenes had been available they would probably have been satisfactory. For these reasons, nitrobenzene and trimethyl-benzene were

dropped from further consideration. Reproducible results were obtained when using toluene, so further work was confined to this solvent. Inasmuch as alumina did not operate well with toluene, the remaining alternative (with due consideration for both the chromatograph separation and the optical analysis) was the combination of basic copper carbonate with toluene.

The optical densities of pure benzanthrone and pure 2:2'-dibenzanthronyl in toluene are given in Table 2 and are plotted in Figure 2. The average of two determinations was taken for each value.

JLNB-4308, pages 33, 34, 41, 42.

Table 2

Wave Length (Millimicrons)	Optical Densities of Benzanthrone in Toluene (.01 mg./ cc. 1 cm. cell.)	Optical Densities of 2:2'-Dibenzanthronyl in Toluene (.01 mg./ cc. 1 cm. cell)
400	.3871	.6070
405	.3338	.6328
410	.2351	.6566
415	.1284	.6174
420	.0612	.4905
430	.0086	.2221
440	.0000	.0753
450		.0199
460		.0055
470		.0028

The curves of Figure 2 indicated that a quantitative simultaneous determination of the two components might be worked out based upon the simultaneous linear equations.

$$a_1X + b_1Y = c_1$$
$$a_2X + b_2Y = c_2$$

where X and Y are the concentrations (fractions of .01 mg. per cc.) of dibenzanthronyl and benzanthrone respectively, and a_1, a_2 are the densities of pure dibenzanthronyl at 410 and 400 millimicrons; b_1, b_2 are the densities of pure benzanthrone at 410 and 400 mm; and C_1, C_2 are the observed densities of a sample possibly containing a mixture of the two bodies. These equations merely state than an observed density (such as C_1) is the result of the sum of the densities of the two components at that particular wave length. These equations hold only in case the system follows Beer's law which

FIGURE 2

OPTICAL DENSITIES OF

PURE BENZANTHRONE AND PURE 2,2'-DIBENZANTHRONYL

IN TOLUENE

.01 Milligram per cc 1cm cell

REFLECTANCE (PERCENT)
TRANSMITTANCE
OPTICAL DENSITIES

1.00
.90
.80
.70
.60
.50
.40
.30
.20
.10
0

2,2'-Dibenzanthronyl

Benzanthrone

400 20 40 60 80 500 20 40 60 80 600 20 40 60 80 700

WAVELENGTH IN MILLIMICRONS

TEST NO. _____ BY _____

DATE _____

INSTRUMENT NO.

GENERAL ELECTRIC

RECORDING SPECTROPHOTOMETER

DUP050041085

states in effect that the optical density is directly proportional to the concentration of the solute. This is tested for later. The terms a_1X - - etc. stand for the optical densities of the individual components at a particular wave length. Referring to Table 1, and using the above equations, we find that

a_1 = density of pure dibenzanthronyl at 410 millimicrons = .6566
a_2 = density of pure dibenzanthronyl at 400 millimicrons = .6070
b_1 = density of pure benzanthrone at 410 millimicrons = .2351
b_2 = density of pure benzanthrone at 400 millimicrons = .3871
c_1 = observed density (corrected to standard conditions) at 410
c_2 = observed density (corrected to standard conditions) at 400

Inserting these values in the equations and solving for X and Y (the concentrations of dibenzanthronyl and benzanthrone expressed as decimal fractions)

$$X = 3.472\ c_1 - 2.109\ c_2$$
$$Y = 5.889\ c_2 - 5.444\ c_1$$

Known mixtures of pure benzanthrone and pure 2:2'-dibenzanthronyl were then made up in toluene and examined in duplicate by this method. Each result is the average of two determinations. The results are tabulated in Table 3.

Table 3

	Composition %			
	Known		Found	
Reference	Benzanthrone	2:2'-Dibenz'l	Benzanthrone	2:2'-Dibenz'l
4308-45	4.97	95.03	4.94	93.9
4308-56	4.98	95.02	4.7	95.4
4308-57	41.99	58.01	40.8	57.6
4308-58	42.03	57.97	41.3	58.3

Thus on the optical end, the method appeared to be accurate to less than 1% over a wide range of compositions, indicating that the system follows Beer's law.

The efficiency of the chromatograph tube was tested as follows: Known samples of pure 2:2'-dibenzanthronyl were made up in toluene and passed through tubes made up with basic cupric carbonate followed by a toluene wash. The results of duplicate runs are given in Table 4.

Table 4

Reference	Optical Density at 410 millimicrons of .01 mg. per cc. in 1 cm. cell Known	Found	Efficiency
4367-81	.6566	.6538	99.57%
4367-82	.6566	.6510	99.15%
			99.36% Average

Thus there appears to be an average loss of 0.64%, and the method may therefore possibly be in error by that amount. In running these blank tests it was found that the amount of wash had a great influence on the accuracy of the results. With 50 cc. of toluene wash there was wide variance in the results and they were of course less than the true value. Results which could be duplicated to 1% or less were not obtained until the wash was increased to 100 cc. These experiments were carried out with 10 milligrams of sample in 75 cc. of toluene. With larger samples, the amount of wash would have to be increased.

Application:

For a detailed account of the method see the Appendix. This method was applied in analyzing some representative samples of crude and purified 2:2'-dibenzanthronyl, and a residue from a purification. The results are given in Table 5.

Table 5

2:2'-Dibenzanthronyl Sample	Analysis Dibenzanthronyl	Benzanthrone	Ash	Oxy Bodies + Dibenzanthrone	Reference
Plant Crude	71.4	3.0	1.19	24.4	4308-65
	71.6	2.7	1.19	24.5	
Purified	93.9	0.0	.38	5.7	4308-153
	93.4	1.8	.38	4.4	
Residue	41.1	4.9	.66	53.3	4308-136
	40.0	5.2	.66	54.8	

Analysis of Residue:

The alkaline pyridine bases filtrate from the purification of crude 2:2'-dibenzanthronyl described in JLR-33-45, No. 1, Serial Number 18165, contains a residue which is isolated by steam distillation. It was found that the recommended analytical method could be applied to this material if it was first treated with acid, as by an acid pasting from sulfuric acid, followed by washing acid-free and drying. Without this preliminary treatment trouble is caused by lack of complete adsorption of some of the impurities in the chromatograph column. This was possibly due to traces of high boiling pyridine base residues which decreased the adsorption. In Table 5 the third item (Reference 4308-136) was treated by this method. The difficulty might also possibly be overcome by running through a second column, but this was not tried.

References:

U.S. 1,564,423; Luttringhaus to Badische, 1925
JLR-33-45, No. 1, Serial Number 18165.
JLR-33-45, No. 3, Serial Number 18808.
I.C.I. Report H-3520.
Patent Report AN-2336
I.G. Plant Process for 2:2'-Dibenzanthronyl, August 1932
Luttringhaus and Neresheimer; Ann. 473 273 (1929)
JLR-33-50, No. 1, Serial Number 18733.

Appendix:

The following method is recommended for the analysis of 2:2'-dibenzanthronyl:

Equipment Required

Weighing bottle
Howell flask, 125 cc. See Figure 1-A of the text. Made by cutting the rim off of an Erlenmeyer flask and molding on a pouring spout.
Chromatograph tube. See Figure 1-B of text.
Filter flask, 250 cc.
Volumetric flask, 250 cc.
Volumetric flask, 100 cc.
Spectrophotometer reading to 400 millimicrons.

Materials Required

Basic cupric carbonate. Ground not finer than about 10 microns. Supplied by J. T. Baker Chemical Co., Phillipsburg, N. J.
Toluene. Dry, water white redistilled.

Procedure:

Dry the sample of 2:2'-dibenzanthronyl at 110°C. and grind it to pass a 60 mesh screen. Weigh out about 10 milligrams to an accuracy of .0001 gram. Transfer the sample to the Howell flask, washing out the weighing bottle with three small portions of toluene from a 75 cc. supply. Then add the rest of the toluene to the flask. Heat the mixture just to the boil, crushing any undissolved particles with a glass rod. (There will be a small amount of insoluble residue due to ash). Allow the solution to cool to room temperature with occasional stirring. While the solution is cooling, prepare a chromatograph tube as shown in Figure 1-B. A calcium chloride tube is used for this purpose, the bulb of which is filled with glass beads. A layer of cotton is pressed down next, followed by the copper carbonate, which forms a column about 15 mm. in diameter by about 55 mm. high. Pour 10 cc. of toluene into the tube and suck down until almost to the surface of the copper carbonate. When the toluene solution has cooled to room temperature, run it through the tube using vacuum on the flask, and finally wash out the Howell flask with three small portions of toluene, adding them to the chromatograph tube, and finally, run 100 cc. of toluene through the tube. Pour the eluent and wash into a tared 250 cc. volumetric flask, and weigh to 0.1 gram. Shake well. Pipette a 20 cc. portion into a tared 100 cc. volumetric flask, weigh to 0.01 gram and make up to the mark with toluene. Reserve a 50 cc. sample of the same toluene for the blank cell of the photometer.

Measure the percent transmission of the final solution in the spectrophotometer at 400 and at 410 millimicrons, and convert these readings to optical density by the formula $D = \log 1/T$, and correct the so-obtained observed density values to standard conditions of 1 cm. cell thickness and concentration of 0.01 mg. per cc. The correction factor is obtained as follows:

$$F = \frac{1}{\frac{w_1 w_3 t}{w_2}}$$

where w_1 = weight of sample in milligrams
w_2 = weight of eluent and wash in grams
w_3 = weight of aliquot in grams
t = cell thickness in cm.

The corrected density readings are inserted in the formulae

$$X = 3.472\ d_2 - 2.109\ d_1$$
$$Y = 5.889\ d_1 - 5.444\ d_2$$

where X = the purity of the 2:2'-dibenzanthronyl as a decimal fraction
Y = the proportion of benzanthrone present as a decimal fraction.
d_1 = corrected observed density at 400 millimicrons
d_2 = corrected observed density at 410 millimicrons

The following typical determination illustrates the method.

Weight of sample (w_1) - 10.7 milligrams
Weight of eluent (w_2) -192.15 grams
Weight of aliquot (w_3) - 17.20 grams
Thickness of cell (t) - 1.0057 cm.

$$\text{Factor} = \frac{1}{\frac{10.7 \times 17.20}{192.15}} \times 1.0057 = 1.0381$$

Wave length	T	d(obs.)	d(std.)
400	28.0	.5530	.5741
410	25.4	.5950	.6177

X = 3.472 x .6177 - 2.109 x .5741 = .9339 = 93.4% dibenzanthronyl purity
Y = 5.889 x .5741 - 5.4444 x .6177 = .0181 = 1.8% benzanthrone

Notes and Precautions:

1 - If basic cupric carbonate finer than about 10 microns is used, the rate of flow is decreased to such an extent that the method takes too long a time.

2 - Great care must be taken in weighing the sample since an error of .0001 gram in weighing introduces a 1% error in the results. This could be eliminated by taking a larger sample, but at the sacrifice of the convenient small volumes.

3 - The equipment must be scrupulously clean and dry. Color taken up by the solutions from foreign matter may radically affect the spectrophotometric readings. The presence of moisture or alcohol may interfere with the proper adsorption of the impurities.

4 - Duplicate determinations should check each other to within about 1%.

5 - All calculations should be done by machine or by five place logarithms, as the slide rule is not accurate enough.

Submitted for typing - June 21, 1944
Typed - June 22, 1944.
emh