

E. I. DU PONT DE NEMOURS & COMPANY
KREBS PIGMENTS DEPARTMENT
256 VANDERPOOL STREET
NEWARK, NEW JERSEY

NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

Final Report

Title: REACTIVITY OF RT-551-D, LABORATORY
90% 5-Nitro-2-Amino-Anisole → "Naphthanil" ASD
10% 5-Nitro-2-Amino-Anisole → BON, Manganese

Period Covered: FEBRUARY 1946 to JUNE 1948

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SUBMITTED BY: A. D. MILLS

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APPROVED BY: A. A. BRIZZOLARA

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INTRODUCTION:

RT-551-D(A), K-2292 (5-nitro-2-amino-anisole → "Naphthanil" ASD) is a durable automotive maroon for use in alkyd systems but has severe reactivity and thixotropy upon aging in the can. A study was undertaken in the laboratory to eliminate, if possible, these undesirable rheological properties. During the course of the work several distinct lines were developed, which will, of necessity, have to be reported separately. In this report the several developments will be correlated and the general results summarized.

There were two lines of thought concerning the approach and both were considered simultaneously. These were variations in the coupling per se (variable study) and a study of the effect of possible impurities such as "Lithosol" Acid BON, ortho toluidine, the soda salt of BON and N-ASD, the coupling of 5-nitro-2-amino-anisole → BON, nitrosamine salt and decomposed diazo.

SUMMARY AND CONCLUSIONS:

1. The adding of the aforementioned possible impurities to RT-551-D(A) in 10% amounts resulted in two leads. One was the use of 5-nitro-2-amino-anisole → BON, Manganese and the other decomposed diazo. Decomposed diazo gave results equivalent to the BON manganese product but because of the unstable nature of these decomposition products the work on the BON coupling material (RT-571-D) was stressed.

When this latter material was blended with RT-551-D(A) in a 10/90 ratio, the reactivity in alkyds was greatly reduced. This led to the development of RT-551-D(D) which is a blend of 90 parts of RT-558-D(B) (originally RT-551-D(A)) and 10 parts of RT-571-D(C) and is practically non-reactive in GE-2458. Other amounts of the BON, Mn additive, from 5 to 25%, were used but the 10% amount gave the greatest effect without detracting from the durability. The complete laboratory, semi-works and plant developments are covered in KN-49-8, KN-49-9, and KN-49-10.

2. Modifying the coupling procedure of the 5-nitro-2-amino-anisole → NASD by the usual variables did not reduce the reactivity at all. However, a product lighter tinctorially was obtained which realized a savings of about 20 cents per pound and was coded K-2413. The work leading to this formula will be covered separately in KN-49-11.

3. Various materials, such as metal oxides and hydroxides and other pigments made from BON and laked with manganese, were added to RT-551-D(A) but with no significant results.

4. The procedure of co-coupling the BON and NASD followed by laking with manganese was attempted and in every case the reactivity was increased.

5. Both semi-works and plant batches of RT-588-D and RT-571-D were evaluated in the laboratory and the resulting mixes were submitted to the plant for blending into RT-551-D. The actual blending of the semi-works and plant products is covered in KH-49-7 and DSN-49-9.

GENERAL DISCUSSION:

In addition to the manganese-BON and decomposed diazo additives, the following materials were added to RT-551-D(A): nitrosamine salt, the soda salts of NASD and BON, manganese-BON, $\text{Mn}(\text{OH})_2$, CaO , ZnO , MnO_2 , "Ti-Pure" R, Timinox, Kadox, alumina hydrate, $\text{Ca}(\text{OH})_2$, the decomposition products of para-nitraniline, meta-nitro-para-toluidine and para-chlor-ortho-nitraniline, RT-551-D, RT-525-D, RT-499-D, RT-436-D and K-2405. Several, such as Kadox and Timinox, reduced the reactivity but so distorted the color as to be of no practical value.

Considerable work on a variable study of RT-551-D(A) was done but nothing reduced the reactivity within the desired tintorial range. In most cases the undeveloped products were superior in reactivity to the developed but the color too light. The presence of BON was believed to be contributing to the reactivity of RT-551-D but several attempts were made to dissolve purified "Naphthanil" ASD at room temperature with the aid of wetting agents but no change in reactivity was recorded. Also a coupling was made in an alcohol medium to eliminate conversion to BON but no advantage was shown.

Attempts were made to reduce the reactivity of RT-551-D by coupling with a deficiency of NASD, coating with sodium methyl silicolate, treating presscake with toluene and treating slurries with calcium rosinate and magnesium naphthenate but none improved the reactivity.

Regarding the actual blending to prepare RT-551-D(D), all the semi-works and plant lots of RT-571-D and RT-588-D (K-2292) were pre-blended in the laboratory and evaluated in alkyd (GE-2488) for color and viscosity. As these blends constituted plant development of RT-551-D, a separate report will cover this phase.

CLARIFICATION OF CODES:

As the Sales Division desired that the RT-551-D code be used for the sale of the finished product, it was necessary to give the "Naphthanil" coupled product a new code (RT-588-D) and RT-551-D became a mix. The developments of the codes is listed below.

RT-551-D(A) = the original code on 5-nitro-2-amino-anisole + "Naphthanil" ASD. It was also known as K-2292 and is now coded RT-588-D.

RT-588-D - is the present code for the straight
"Naphthanil" coupling formerly RT-551-D(A).

RT-571-D (A,B & C) - RT-571-D always refers to the
additive 5-nitro-2-amino-anisole → BON,
manganese. Originally was coded K-2384.

RT-551-D (B,C & D) - 90/10 blends with RT-588-D and
RT-571-D (A,B & C).

EXPERIMENTAL DETAILS:

Evaluations were made in the alkyd vehicle GE-2458 in
the following proportions.

17 g. Pigment (15.5 g. RT-588-D + 1.5 g RT-571-D)
72 g. GE-2458
9 g. Union 30

Ground 48 hours.

80 g. GE-2458
9 g. Union 30
2.2 cc. KV-30

The experimental references for:-

(1) the variables covered in detail in this report are:-

<u>N.B.</u>	<u>Exp.</u>		<u>Page</u>
1197	3	Preparation of samples believed to be impurities.	15
"	4	Evaluation of 1197-3.	18
"	5	Temperature of Coupling Study.	21
"	6	Preparation of decomposed diazo.	25
"	7	Use of additional caustic in coupling.	27
"	8	Temperature of coupling variable in K-9942.	30
"	9	Repeat evaluation of 1197-3.	33
"	20	Reduction in xs NASD, NaHCO ₃ & volume.	88
"	21	Study use of wetting agents with NASD.	92
"	22	Use purified NASD & DuPont NA.	94
"	23	Study pH of coupling of K-2292.	98
"	24	Study of amount of hydrolysis of NASD.	101
"	25	Treating developed slurries with HCl, NaOH & Na ₂ CO ₃ .	107
"	26	Study pH of development.	116
"	29	Variation in temperature of coupling.	120
"	31	Addition of various manganese-BON Toners.	128
"	35	Rebut of RT-551-D vs. Harmon's MT-200.	146
"	43	Preparation of Mn-BON & Mn(OH) ₂ .	170
"	49	Addition of K-2405 to K-2292 in 5-25% amounts.	190

<u>N.B.</u>	<u>Exp.</u>		<u>Page</u>
1245	2	Addition of various metal oxides & hydroxides.	11
"	8	Preparation of large sample of K-2054.	32
"	34	Couple K-2292 with a deficiency of NASD.	111
"	35	Preparation of large sample of decomp.diazo.	116
"	36	Use of sodium methyl silosolate on K-2292.	118
"	49	Repeat of 1245-34.	161
"	50	Repeat of 1245-36.	165
"	54	Treatment of presscake with toluene.	182
1264	5	Coupling in an alcohol medium.	21
"	11	" " " " " "	38
"	13	Eval. of crude 5-NO ₂ -2NH ₂ -An.	47
"	23	Substitution of BN, BON, Mono Acid F & R Salt for 5% NASD.	81
"	26	Rebut of 1197-22A ₂ & Bg.	92
"	35	Substitution of CaCO ₃ for NaHCO ₃ & use of calcium rosinate and magnesium naphthenate.	120

(2) All the work on this problem including the development of RT-571-D, K-2413 and the blending of plant lots is covered in the following notebooks.

1197 - complete
1245 - complete
1264 - complete
1302 - Exp. #3

REFERENCES:

KN-49-7 - Plant Blending of RT-551-D.
KN-49-8 - Laboratory Development of RT-571-D.
KN-49-9 - Semi-Works " " " "
KN-49-10- Plant " " " "
KN-49-11- Laboratory Development of K-2413.
KN-49-12- Semi-Works " " " "
DSN-49-9- Plant Blending of RT-551-D.