Diamond Alkali Company

JNTER-OFFICE CORRESPONDENCE
DACO 118

DATE
November 9, 1954

TO
Mr. J. J. Burton

FROM
Mr. E. Hofgesang

SUBJECT: Isopropyl 2,4-D Ester Manufacture

cc: K. Aspinwall
J. R. Trocki
Lab. File

PURPOSE OF INVESTIGATION:

1. To evaluate toluene as a substitute for benzene in our present plant process.

2. To determine what can be done to shorten the reaction time per batch of material.

3. To determine if the present plant benzene usage per batch can be reduced.

SUMMARY OF RESULTS:

1. Toluene can be used in place of benzene in our present plant process on the same weight basis.

2. The reaction time required for esterification can be reduced by first removing the water from the wet 2,4-D acid.

3. The benzene charge could be reduced in the laboratory by one half the present charge without affecting the reaction.

GENERAL METHOD OF INVESTIGATION:

The method employed is primarily intended to keep all reaction variables constant. A typical plant batch of 2,4-D acid was selected (containing 12% H_2O) and used throughout the entire investigation. The heat input per unit time was kept constant for all reactions. The drying and washing procedure was the same in all cases.

The equipment consisted of a one liter flask, a separator for water removal, water condenser, and a thermometer suspended through the condenser with the thermometer bulb coming in contact with the vapor just before entering the condenser. The vapor temperatures for the initial phase of the investigation (using toluene) were measured incorrectly and therefore, are omitted. In all experiments using benzene the vapor temperatures ranged between 67-70°C. Heat was supplied with a heating mantle controlled by a rheostat.

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DATA AND RESULTS:

The use of toluene as a substitute for benzene was conducted in the following manner. Two esterification reactions were run, one reaction using the present plant raw material figures and the second substituting toluene in place of benzene on the same weight basis. Both reactions were considered esterified at the end of 2 hours and 40 minutes by having an acid value less than 10. The reaction using present benzene plant charges had an acid value of 7.2, while the reaction using toluene had an acid value of 8.1, the difference being insignificant. The finished ester (using toluene) had a specific gravity of 1.270 at 20°C., an acid value of 1.4. No odor of toluene could be detected in the finished ester. There was no usual difference in color.

The advantages of using toluene as a substitute for benzene are as follows:

1. Saving of approximately 6¢ per gallon.

2. Lower toxicity.

3. Lower vapor pressure resulting in lower losses.

At the present time, the only disadvantage which can be realized, although not proven, in the laboratory may be a difference in color of the finished ester. The use of toluene requires a reaction temperature from 10-30°C. higher and on a plant scale the temperature would be maintained for a longer period of time than in the laboratory, which would favor any tendency to darken.

Three esterifications were run all using the present plant charge ratios:

	Lab. Charge
3500 lbs. 2,4-D acid (dry basis).....	221 gms.
300 gals. benzene	139 gms.
200 gals. isopropyl alcohol	83.2 gms.
2 gals. conc. H_2SO_4	2.0 gms.

The first (#1) esterification was run according to plant procedure whereby the alcohol and benzene are added to the 2,4-D acid. The sulphuric acid is added and the material is then esterified.

Two runs (#2 and #3) were performed, whereby the benzene was added to the 2,4-D acid and the major portion of the water azeotroped off. The isopropyl alcohol and catalyst were then added and the reaction continued until completion (acid value less than 10) which required approximately 40% less time than the present procedure (#1).

	#1	#2	#3
Total time required for reaction	2 hrs. 40 min.	1 hr. 40 min*	1 hr. 30 min.*
Acid value at completion or reaction	8.2	9.1	9.0

*NOTE: Included, is the time required to remove the water from the acid, which is 25 min. and 22 min. respectively.

The present plant ratio of benzene to isopropyl alcohol is 300/200 gallons. Four laboratory runs were performed to determine the effect of a higher or lower benzene ratio. The following benzene isopropyl alcohol ratios represent figures which were respectively reduced to a laboratory scale. The reactions were performed using the present plant procedure.

Plant Ratios (Proportionately reduced to a laboratory scale)	Esterification Time	Final Acid Value
150/200 gals. benzene isopropyl alc.	2 hrs. 38 min.	8.5
200/200 " " " "	2 " 35 "	8.3
300/200* " " " "	2 " 30 "	7.2
400/200 " " " "	2 " 35 "	8.9

*Present ratio

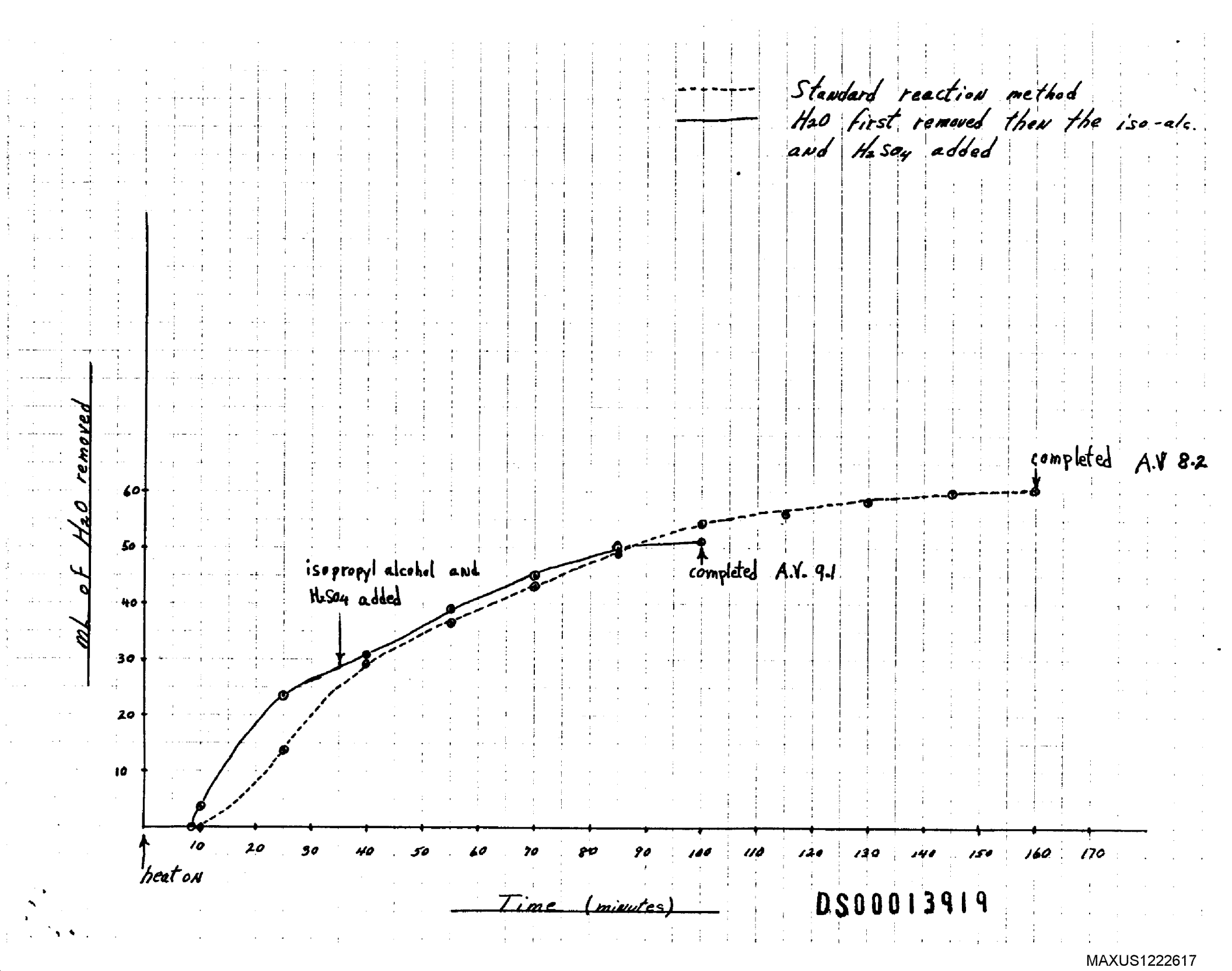
In the laboratory, there appears to be no significant reaction time difference, by reducing the present benzene charge by one half.

RECOMMENDATIONS:

1. Various methods should be investigated for removing the H_2O from the 2,4-D acid prior to esterification.

E. Hofgesang

EH:jp



The differences in the quantity of water collected during the reaction can be partially explained by this specific gravity

Standard Reaction method — .977 @ 20°C
Jaco Method .989 "

This would indicate that by removing the water first from the wet 2,4-D acid a saving of isopropyl alcohol can be effected