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SPECIAL APPLICATION REPORT

ON

PLASTICIZER SALES DEVELOPMENT RESEARCH - 1955

PART I: THE MONTARS

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## INTRODUCTION

In the production of the Aroclors and Santowax R, residues from the distillation phase have been collected and then discarded. These residues, named Montars, have been assumed numbers (1 through 10) more or less corresponding to their parent compounds. Table I. Chemically, the Montars are polychlorinated polyphenyls. Their chlorine content varies from 28 to 57% and their calcium oxide content from 0.8% to 7%.

In 1953 the average monthly production of Montars was 182,000 pounds with Nos. 1, 3, 4, and 5 accounting for 60% of that amount.

At the present time the Anniston plant pumps the residues to a dump while the WGK plant must drum and haul them to a dump. The cost of these methods of disposal is estimated at 0.25 to 1.2 cents per pound. Mention should be made then of the dollar advantage to Monsanto of selling these compounds, in preference to the present cost of disposal.

## SUMMARY

The Montars were tested as replacements and extenders for asphalt in adhesives and other asphalt applications; as fire retardant additives to asphalt formulations, to polyesters and to polyvinyl chloride formulations.

Laboratory test results indicate that the Montars have application possibilities in each of the above categories.

Because Montar No. 9, a residue from Santowax R, does not contain chlorine, it was not included in the tests.

## CONCLUSIONS

Laboratory test data indicate that:

1. The Montars, if sold at about \$0.05 per pound, are the most inexpensive source of organic-combined chlorine presently available. Table III. Other organic-combined chlorine compounds sell in the 16-18¢/lb. range.
2. The Montars are soluble in most aromatic solvents and are especially soluble in the kerosene-type solvents. Table IV.
3. The Montars are compatible with asphalt up to two parts of Montar to one part of asphalt.
4. They impart flame retardancy to asphalt. Table V.
5. The Montars were found to be compatible in polyvinyl chloride formulations up to a concentration of 10 PHR (10 parts per hundred parts of resin).

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6. They are compatible in polyester formulations and impart some flame retardancy to those formulations.

#### RECOMMENDATIONS

Montars should be stressed as inexpensive sources of organic-combined chlorine, as extenders and replacements for asphalt and as flame retardant additives to asphalt, polyvinyl chloride and polyester formulations.

This information could well be incorporated into a technical bulletin on the Montars and their application.

#### PATENT STATUS

Items of a patentable nature have been precluded by early patents covering the application of chlorine-containing organic compounds.

#### EXPERIMENTAL

Production of the Montars, being geared to that of the Aroclors and Santowax R, will vary from month to month, and as a result, no specific amount is produced each month.

The figures in Table I, though for the year 1953, held true for 1954 with the exception of a decrease in No. 4 and a like increase in No. 1.

TABLE I  
AVERAGE MONTHLY PRODUCTION OF MONTARS  
IN 1953

| <u>Montar</u> | <u>Parent Compound</u>           | <u>Percent Residue</u> | <u>Pounds Produced</u> |
|---------------|----------------------------------|------------------------|------------------------|
| 1             | Aroclor 1242                     | 4                      | 25,600                 |
| 2             | Aroclor 1248                     | 4                      | 4,000                  |
| 3             | Aroclor 1254                     | 4                      | 20,000                 |
| 4             | Aroclor 1260                     | 4                      | 28,000                 |
| 5             | Aroclor 5460                     | 15                     | 45,000                 |
| 6             | Aroclor 4465                     | 8                      | 320                    |
| 9             | Santowax R                       | 15                     | 60,000                 |
| 10            | Mixture of No. 5 and No. 9 (1:1) |                        | 182,920                |

The physical properties of the Montars in Table II show the Montars to have a wide range of properties relative to their boiling points, their physical appearance and their flash and fire points.

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TABLE II

PHYSICAL PROPERTIES OF THE MONTARS\*

| Montar | Appearance  | Softening Point | Flash Point | Fire Point     | Penetration                           |
|--------|-------------|-----------------|-------------|----------------|---------------------------------------|
| 1      | brown solid |                 |             |                |                                       |
| 2      | brown solid |                 |             |                |                                       |
| 3      | brown solid | 120-170°C       | 298°C       | None at 343°C. |                                       |
| 4      | brown solid | 140-200°C       |             |                | None at 75°C.                         |
| 5      | black solid | 180-250°C.      |             |                | $2 \times 10^{-2}$ cm/min. at 75°C.   |
| 6      | black solid | 150-210°C       |             |                |                                       |
| 9      | black mass  | 130-160°C       | 257°C       | 304°C.         |                                       |
| 10     | black solid | 140°C.          |             |                | $47 \times 10^{-2}$ cm/55 cc at 75°C. |

\*Data obtained from Montar Data Sheet

The salient feature of the data in Table III below is the quantity of organic chlorine to be found in most of the Montars. To the best of this writer's knowledge, they are the least expensive source of organic-combined chlorine available.

TABLE III

TYPICAL CHEMICAL ANALYSIS

| Montar | Chemical Name              | % Chlorine | % Calcium Oxide | % Iron |
|--------|----------------------------|------------|-----------------|--------|
| 1      | polychlorinated polyphenyl | 40.5       | 0.12            | 0.07   |
| 2      | polychlorinated polyphenyl | 46.2       | 1.8             |        |
| 3      | polychlorinated polyphenyl | 51.6       | 3.5             | 0.31   |
| 4      | polychlorinated polyphenyl | 55.4       | 6.9             |        |
| 5      | polychlorinated polyphenyl | 57-59      | 4.5             | 0.12   |
| 6      | polychlorinated polyphenyl | 58.6       | 2.0             | 0.13   |
| 9      | polyphenyl                 | 0          | 0.8             | 0.57   |
| 10     | mixed No. 5 and No. 9      | 28         | 2.5             | 0.06   |

Solution data on the Montars show them to be very soluble in aromatic solvents and soluble in kerosene-type solvents. The ease of solution should make the Montars valuable in replacing asphalt in applications such as use in adhesives.

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**TABLE IV**  
**SOLUTION DATA**

| <u>Montar</u> | <u>Aromatic*</u> | <u>Kerosene Type</u> | <u>Methanol</u> | <u>Acetone</u> |
|---------------|------------------|----------------------|-----------------|----------------|
| 1             | VS               | S                    | SS              | VSS            |
| 2             | VS               | S                    | SS              | VSS            |
| 3             | VS               | S                    | SS              | VSS            |
| 4             | VS               | S                    | SS              | VSS            |
| 5             | VS               | VS                   | SS              | SS             |
| 6             | VS               | VS                   | SS              | SS             |
| 9             | VS               | SS                   | I               | SS             |
| 10            | VS               | SS                   | I               | SS             |

\*Such as benzene or toluene.

KEY

|     |                       |    |              |
|-----|-----------------------|----|--------------|
| I   | Insoluble             | S  | Soluble      |
| VSS | Very Slightly Soluble | VS | Very Soluble |
| SS  | Slightly Soluble      |    |              |

EXPERIMENTAL - Potential Application

A. In Adhesives

A 50% solution of Montars 2, 5, and 4 in Velsicol 50 mixed 1:1 with a 60%-solids, rubber emulsion provided good tack when tested as a paper adhesive. The tack appeared to be as good as that obtained when using a 50% solution of asphalt in kerosene. Other Montars, tested in similar fashion, gave comparable results.

To 25 g. of the above Velsicol solution, 1.25 g. and 2.5 g. of an emulsifier was added. These mixtures were then diluted with water. The sample containing 5% emulsifier remained in an emulsified state until it was diluted 160% with water. The one containing 10% emulsifier did not break until diluted 30% with water.

These test results tend to show that the Montars could replace or extend asphalt in the adhesive field at a saving to the formulator, because they provide equivalent tack, are equally soluble and emulsify well.

B. As a Flame-Retardant Additive

Varying amounts of Montar 4, ranging from 10 to 67%, were mixed on melting with molten asphalt. The flammability of these mixtures were compared to the flammability of asphalt per se. Good flame retardancy was obtained when a minimum of 40% Montar was added to the asphalt.

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Using the stir-in technique, varying amounts of crushed Montar 4 were added to molten asphalt, and the mixture was heated to 190°C. to effect solution of the Montar.

A portion of the mixture was transferred to a crucible and the mixture was heated until the fumes would ignite (approximately 200°C.). The time required for the flames to self-extinguish was then measured. The results follow:

TABLE V  
FLAME RETARDANCY OF THE MONTARS

| <u>Composition</u> | <u>Flame-Out Time</u> |
|--------------------|-----------------------|
| Asphalt, per se    | 210 seconds           |
| 10% Montar added   | 90 seconds            |
| 20% Montar added   | 10-20 seconds         |
| 30% Montar added   | Instantaneous         |
| 50% Montar added   | Instantaneous         |
| 67% Montar added   | Instantaneous         |

The flame-retardant action of the Montar was caused by the formation of an intumescent layer on the surface of the mixture, snuffing out the flame.

This action could be utilized in the production of film and sheeting, such as floor tile, where the limitations of the Montars due to color are not serious.

#### C. As An Extender for Asphalt

The compatibility of Montars in asphalt was determined by mixing and heating the mixtures to effect solution. Montars are readily compatible at least up to the ratio of 2 parts Montar to one part asphalt.

It was noted that as the concentration of Montars increased the softening point and the brittleness of the mixture also increased.

#### D. As A Flame-Retardant Additive to Polyesters

Montars 2, 4, and 6 were added to a series of polyester formulations with the Montar concentration being 1%. These formulations were then molded into 1 inch squares which were suspended above a flame and the flammability visually noted.

The polyesters with Montars were considered to be flame-retardant when compared to polyesters not containing any of the Montars.

This application is again limited by the inherent color of the Montars.

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6.

#### DISCUSSION

Since the color of Montars is dark brown to black, their use in vinyls, in adhesives and in polyesters is limited to those applications where the color of the Montars is of little importance.

However, with an estimated selling price of 5¢ per pound, the Montars should certainly be attractive as an inexpensive source of chlorine, as an extender or replacement for asphalt and as a flame-retardant additive.

#### ACKNOWLEDGMENT

The writer is indebted to Dr. A. Martin Ellenburg for production and physical and chemical analysis data and to Mr. C. F. Bourbeau for tests run on Montars in polyesters.

#### APPENDIX

Laboratory notebook pages 58451 and 58452 in Book No. A-1170 issued in 1954 to R. M. Parks.

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