PLAINTIFF'S EXHIBIT UC-802

WV-0008 # 6
Sullivan

SUMMARY REPORT

# HEALTH HAZARD OF INSULATION MATERIAL
# DETERMINATION OF ALPHA-QUARTZ BY X-RAY DIFFRACTION

AUTHORS: H. E. Persinger
K. J. Garska

DATE: April 4, 1962

FILE NO: 244M-F1

Project No: 9250X

SUMMARY Two materials being used for thermal insulation at the Institute Plant have been determined by X-ray diffraction to contain a considerable amount of asbestos and only a small amount of alpha-quartz. High concentrations of either alpha-quartz or asbestos in the dusts produced when handling these materials would constitute a health hazard to personnel applying them.

INTRODUCTION A request was made by Mr. R. E. Peele, the Industrial Hygienist for the Institute Plant, to investigate two insulation materials, Kaylo and Thermo-Bestos, in an effort to determine the amount of alpha-quartz in each. Kaylo is supplied by Owens Corning and Thermo-Bestos is supplied by Johns Manville, Inc. These two insulators produce dusting during cutting; hence, knowledge of the concentration level of alpha-quartz as well as asbestos in these two materials is required for health hazard classification. The following discussion presents data on the determination by X-ray diffraction of the concentration level of alpha-quartz.

DISCUSSION X-ray diffraction has been used to determine the approximate concentration of alpha-quartz in thermal insulation. The presence of alpha-quartz is determined by comparing the patterns of the materials in question with a reference pattern for alpha-quartz. A standard addition technique is used to obtain semi-quantitative data.

## Qualitative Analysis

Diffraction data for both types of insulation material show that there is a small amount of alpha-quartz in each with the major observable component being some form of asbestos. These

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data are given in Table 1. The presence of alpha-quartz has been established by an increase in the reflection corresponding to the 101 diffracting plane of alpha-quartz when a small amount of alpha-quartz is added to the Kaylo material. The remaining reflections in the two patterns are most certainly due to the asbestos. However, asbestos can exist in several different forms or can consist of a mixture of several components. Thus, it is difficult by X-ray diffraction to determine the exact composition of the asbestos or its concentration. Despite these difficulties, the relative intensities of the asbestos reflections as compared to those of alpha-quartz indicate that two to four times as much asbestos is present.

Quantitative Analysis

A sample of blasting sand was obtained and identified as better than ninety per cent alpha-quartz. The diffraction pattern of the blasting sand matched perfectly with the pattern for pure alpha-quartz, which is reported in the "X-Ray Powder Data File". Two standards were prepared by adding one and five per cent by weight of finely ground blasting sand to the Kaylo material and mixing thoroughly. A calibration curve, shown in Figure 1, was obtained by plotting the peak height of the reflection from the 101 diffracting plane of alpha-quartz against the per cent by weight of alpha-quartz added to the Kaylo material. The curve was extrapolated to zero per cent to complete the calibration.

From the calibration curve thus obtained the weight per cent alpha-quartz in the Kaylo and Thermo-Bestos materials is, respectively 5 and 2.5. The error of this determination may be as much as 10 to 20 per cent of the contained amount, due to orientation effects and diffraction variables that have not been corrected.

NOTEBOOK REFERENCE: D-4251-58
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Attachments:
1 Table
1 Figure

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TABLE I

ALPHA-QUARTZ IN INSULATION MATERIAL
QUALITATIVE DIFFRACTION DATA

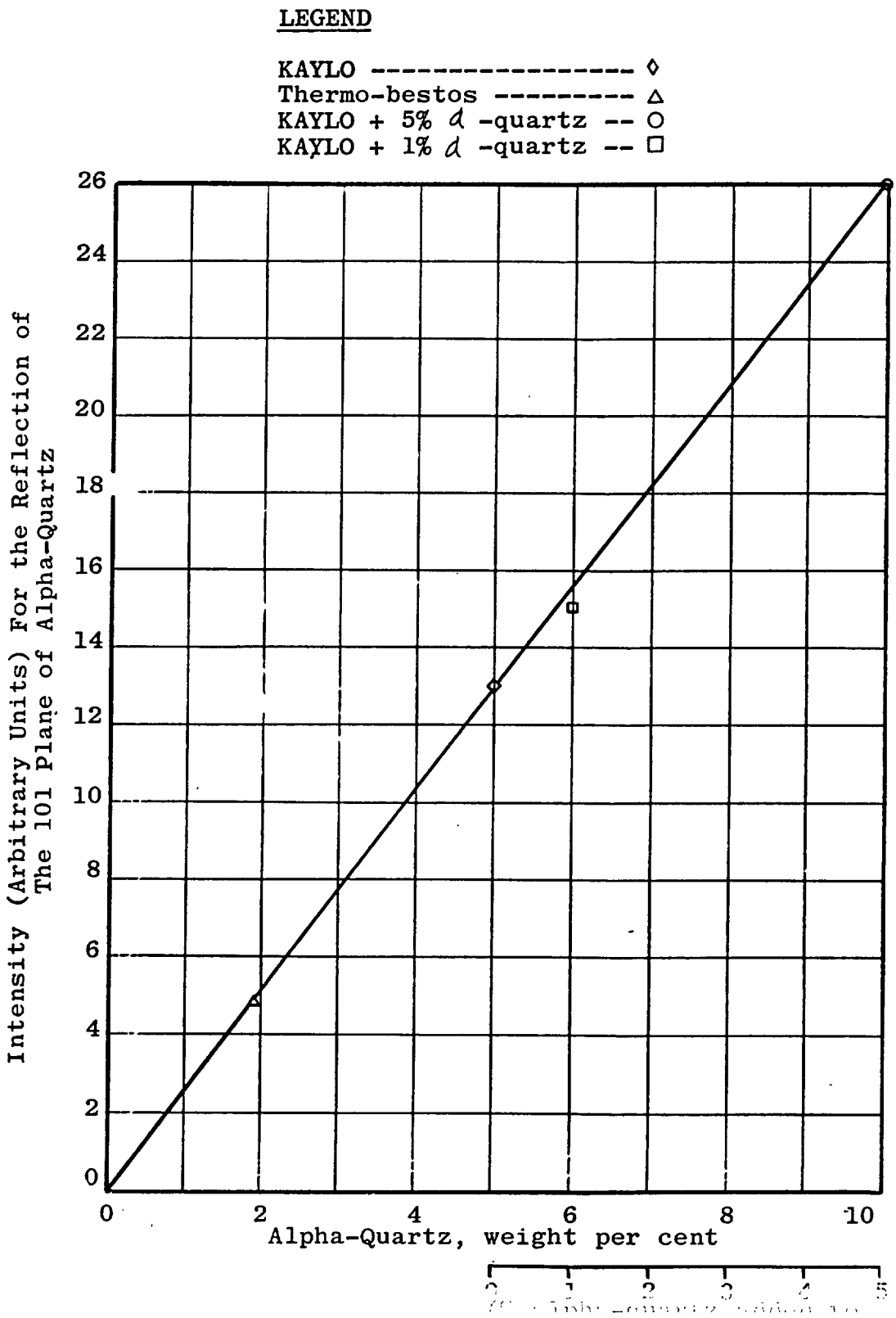
| Kaylo | | Thermo-Bestos | | Alpha-Quartz (a) | |
|---|---|---|---|---|---|
| d-spacing, Å | $I/I_1$ | d-spacing, Å | $I/I_1$ | d-spacing, Å | $I/I_1$ |
| 4.24 | 14 | | | 4.24 | 25 |
| 3.61* | 19 | | | | |
| 3.34 | 62 | 3.33 | 40 | 3.33 | 100 |
| 3.07* | 81 | 3.20* | 25 | | |
| 3.05* | 100 (b) | 3.05* | 100 | | |
| 3.03* | 100 | | | | |
| 2.99* | 52 | | | | |
| 2.796* | 33 | | | | |
| 2.755* | 29 | | | | |
| 2.492* | 29 | | | | |
| 1.835 | 33 | 1.835 | 40 | 1.816 | 12.5 |

(a) There are several more spacings for alpha-quartz that have not been listed. These reflections are too weak to be detected except for material containing about 75 per cent or more alpha-quartz.

(b) Probably represents one diffuse reflection.

* These reflections are due to the components that are generally found in asbestos, i.e. various forms of calcium-magnesium,-aluminum, -etc., silicates.

## ALPHA-QUARTZ IN INSULATION MATERIAL
## STANDARD-ADDITION CURVE





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UC/0021 #6

**SUMMARY REPORT**

# PHENOLIC PIPING

## CHEMICAL RESISTANCE OF UCNC ASBESTOS-PHENOLIC PIPE

AUTHORS: L. S. VanDelinder
D. C. Cook

DATE: July 16, 1963

FILE NO: 1029

Project No: 999G10

SUMMARY Screening tests have been completed to determine the chemical resistance of three asbestos-resinous products prepared by the Union Carbide Nuclear Company. One of these products (No. 766-37) containing a phenolic binder exhibited excellent chemical resistance to a variety of organic and aqueous inorganic solutions. A commercial product based on this formulation should be superior to Johns-Manville Transite and should compare favorably with the J-M Chemtite (phenolic) chemical-resistant piping currently available. Additional laboratory tests and field installation of test sections of the No. 766 formulation will be made.

INTRODUCTION The Union Carbide Nuclear Company is interested in the development of commercial products which might utilize their asbestos product. One of these developments was the possible production of an asbestos-reinforced plastic pipe. Three separate samples of such pipe representing different resinous binders were submitted for evaluation. These had been made into flat stock 1/4" in thickness and were cut to appropriate size for mechanical testing by ASTM D-790 procedures. The three materials were designated as UCNC 465-15, UCNC 640-02, and UCNC 766-37. The resinous binder of the 766-37 was a phenolic resin. This composition has now been given a trade designation of F-100. The matrix of the other two compositions is unknown to the author.

This Department was requested to conduct standard chemical resistance tests of the three materials using the methods developed previously for the evaluation of rigid plastic materials. Test media were selected to represent a broad range of possible exposures with a particular emphasis on streams which might be encountered in waste disposal lines and other common commercial applications.

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In order to obtain an adequate evaluation of the materials, a group of specimens was to be removed after 30, 90 and 180 days. Results of the tests concluded after 30 and 90 days have been reported previously (1). The data obtained by the previous exposures are not included in this report. Only the results of the tests concluded after 182 days of exposure are given in Tables I, II, III and IV.

DISCUSSION Two areas of use of plastic piping of the type developed from the asbestos material are envisioned. One of these is a general application where waste disposal lines, water distribution, hydrocarbon distribution, and other large volume but rather innocuous applications would be involved. These are areas of use where Johns-Manville Transite asbestos materials are currently used in large volume. Consequently, a comparison with the J-M Transite was made by exposure of all materials in dilute acids, alkalis, and certain organic chemicals to roughly approximate the worst conditions anticipated in general piping services.

The data for J-M Transite exposures are shown in Table I and reveal a substantial reduction in the properties of the material after exposure to most all of the test chemicals. In contrast, the No. 465 and 766 materials of the UCNC showed good resistance to essentially all of the chemicals. The 766 material was particularly outstanding in resistance to all test chemicals. The data leave no doubt that the 766 phenolic-asbestos material would be far superior to J-M Transite as a general service piping for miscellaneous service.

The No. 766 product (F-100) is composed of a phenolic matrix and evidenced the usual good chemical resistance of phenolic products. It is interesting to note the resistance of the 766 material to sodium hydroxide. The basic phenolic resin used in this formulation is not resistant to alkalis. The high percentage of asbestos present apparently acted as a sufficient shield to protect underlying layers of phenolic resin and prevented degradation of the product. The use of such a material in alkaline service should be carefully evaluated, however. Alternately, the poor resistance of the product in hydrochloric and sulfuric acid is probably caused by the presence of the asbestos. Chrysotile asbestos is not resistant to inorganic acids and the loss in modulus and other changes noted in the product are probably due to attack by the acids on the asbestos filler.

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(1) Letter of Cook, D. C., to N. J. Setter, Chemical Resistance of Rigid Asbestos, UCNC, March 26, 1963 (ML:500:51).


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The No. 640 material had the poorest overall chemical resistance of the three products with the No. 766 being very definitely superior to all other materials tested.

As stated, the 766 material is superior to Transite as a general purpose piping product. In fact, the material compares most favorably with the present J-M Chemtite phenolic piping material currently offered by that company as a highly chemical resistant process piping. The 766 material has poorer mechanical properties, but the chemical resistance should be roughly the same. The J-M Chemtite contains crocidilite asbestos rather than the chrysotile, and consequently has better mineral acid resistance. However, as a piping material for process work, the 766 would be most attractive where the lower mechanical properties can be allowed.

The poorest property of the 766 (F-100) material is the poor impact resistance. Undoubtedly, the notch sensitivity is very high also. Longer asbestos fibers would be desirable to upgrade these properties, as well as the strength of the product. However, at the present time the F-100 product has strength and modulus in flexure very close to that of the Chemtite product.

Table V shows the appraisal of the various materials tested as determined by an arbitrary limit on changes in weight, dimensions, hardness, strength, and modulus. These limits are entirely arbitrary but have been indicated by the testing of other materials to be significant as limiting changes in the material. No national recognition of these limits for rigid plastic materials is in effect but the values are believed to be accurate for an appraisal of screening tests for such products.

Many types of simple slip fittings may be used on piping made up for a general purpose system. However, when competing with piping materials for process applications, a secure, reproducible, reinforcing fitting must be provided to obtain optimum service from the material. Thus, it is imperative that an excellent fitting be found to join the F-100 piping before it is marketed for process applications. The current J-M molded phenolic fitting is an excellent product, and something of this type is necessary to provide the customer with a complete and usable package.

Additional tests are to be conducted using the F-100 (766) product. The stability of the plastic in a wide variety of organic chemicals will be determined in the manner previously

employed for the evaluation of reinforced plastic materials (2). These data should be available by March, 1964. In addition, various test sections of the piping will be exposed in a variety of plant environments.

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(2) VanDelinder, L. S. and Cook, D. C., Materials of Construction: Evaluation of Rigid Plastics for Organic Chemical Service, February 8, 1962, (ML:500:69-F1).

D. C. Cook

Notebook Reference: 3JCC

ATTACHMENTS
5 Tables

Index: G, 32,112,120,132,155,157,162,166, 169,177,181,190,208,225,242,308

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TABLF I

CHEMICAL RESISTANCE OF J-M TRANSITE

Test Conditions: Ambient Temperature; Static Conditions; 182 days

| Test Media | Spec. No. | % Wt. Chg. | % Dim. Chg. | ASTM D-785-51 Rockwell Hardness "R" Scale | ASTM D-790-58T Flexural Strength, PSI | ASTM D-790-58T Modulus in Flexure, PSI X $10^6$ | Remarks |
|---|---|---|---|---|---|---|---|
| Control | 1 | --- | --- | 114.0 | 8,300 | 3.01 | Unexposed |
| | 2 | --- | --- | 100.0 | 7,890 | 2.98 | " |
| | 3 | --- | --- | 95.0 | 7,090 | 2.71 | " |
| 2% Hydrochloric Acid | 1 | + 8.86 | - 1.29 | 66.5 | 3,980 | 2.41 | Yellow adherent coating |
| | 2 | + 9.61 | + 0.42 | 59.0 | 4,495 | 2.50 | |
| | 3 | +10.48 | nil | 68.0 | 3,765 | 2.08 | |
| 2% Sulfuric Acid | 1 | +21.92 | +12.05 | 63.5 | 4,830 | 2.23 | Loss of binder |
| | 2(1) | --- | --- | --- | --- | --- | |
| | 3 | +16.95 | +11.15 | 72.5 | 3,995 | 2.12 | |
| 2% Nitric Acid | 1 | +14.00 | nil | 74.0 | 2,995 | 3.14 | Adherent coating |
| | 2 | +14.10 | + 0.81 | 89.5 | 4,645 | 2.52 | |
| | 3 | +13.02 | - 0.40 | 95.0 | 3,835 | 2.08 | |
| 2-1/2% Sodium Hypochlorite | 1 | +15.36 | nil | 92.0 | 4,210 | 2.34 | No apparent change |
| | 2 | +14.22 | + 0.41 | 102.0 | 4,155 | 2.30 | " |
| | 3 | + 8.79 | + 0.79 | 110.5 | 4,650 | 3.27 | " |
| 5% Acetic Acid | 1 | + 3.11 | nil | 49.5 | 3,715 | 1.95 | " |
| | 2(1) | --- | --- | --- | --- | --- | |
| | 3 | + 6.67 | nil | 62.0 | 2,970 | 1.60 | " |
| Ethyl Acetate | 1 | + 4.52 | + 0.41 | 101.0 | 4,265 | 1.89 | " |
| | 2 | + 4.13 | + 0.41 | 90.0 | 4,460 | 2.40 | " |
| | 3 | + 1.57 | - 0.81 | 113.0 | 5,480 | 3.25 | " |
| 2% Sodium Hydroxide | 1 | +15.77 | - 0.42 | 96.0 | 4,450 | 2.47 | " |
| | 2 | +15.45 | - 1.22 | 94.5 | 5,110 | 3.10 | " |
| | 3 | +15.80 | + 1.66 | 96.5 | 4,295 | 2.38 | " |
| 2% Ammonium Hydroxide | 1 | +14.30 | - 0.40 | 101.5 | 5,210 | 2.77 | " |
| | 2 | +15.33 | + 0.42 | 101.0 | 4,865 | 3.02 | " |
| | 3 | + 9.88 | - 0.40 | 111.5 | 4,850 | 2.87 | " |
| Kerosene | 1 | +11.35 | nil | 96.0 | 4,420 | 2.73 | No apparent change |
| | 2 | +11.10 | + 0.43 | 99.5 | 4,535 | 2.83 | " |
| | 3 | +11.22 | + 0.41 | 78.0 | 3,170 | 1.74 | " |
| Methyl Isobutyl Ketone | 1 | + 3.95 | - 1.19 | 112.0 | 5,130 | 2.70 | " |
| | 2 | + 4.78 | nil | 82.5 | 3,015 | 2.49 | " |
| | 3 | + 4.57 | nil | 98.5 | 4,250 | 2.25 | " |
| Ethylene Dichloride | 1 | + 6.51 | nil | 94.0 | 4,625 | 2.57 | " |
| | 2 | + 6.98 | nil | 91.0 | 4,125 | 2.28 | " |
| | 3 | + 5.25 | + 1.21 | 106.5 | 4,835 | 2.61 | " |

(1) Specimens used for earlier demonstration and photographs.

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## TABLE II

## CHEMICAL RESISTANCE OF UCNC 465-15

Test Conditions: Ambient Temperature; Static Conditions; 182 days

| Test Media | Spec. No. | % Wt. Chg. | % Dim. Chg. | ASTM D-785-51 Rockwell Hardness "M" Scale | ASTM D-790-58T Flexural Strength, PSI | Modulus in Flexure, PSI X $10^6$ | Remarks |
|---|---|---|---|---|---|---|---|
| Control | 1 | --- | --- | 102.0 | 8,730 | 2.50 | Unexposed |
| | 2 | --- | --- | 102.0 | 10,925 | 2.50 | " |
| | 3 | --- | --- | 103.0 | 10,475 | 2.40 | " |
| 2% Hydrochloric Acid | 1 | - 1.89 | + 0.43 | 89.0 | 6,750 | 1.72 | No apparent change |
| | 2 | - 1.43 | + 0.42 | 92.5 | 8,090 | 1.81 | " |
| | 3 | - 1.54 | + 0.85 | 93.0 | 6,910 | 1.60 | " |
| 2% Sulfuric Acid | 1 | - 2.64 | + 0.85 | 86.5 | 5,890 | 1.62 | " |
| | 2 | - 2.02 | + 0.42 | 88.5 | 6,210 | 1.63 | " |
| | 3 | - 3.53 | + 0.85 | 90.5 | 6,230 | 1.25 | " |
| 2% Nitric Acid | 1 | + 0.69 | + 0.86 | 96.0 | 7,410 | 2.18 | " |
| | 2 | + 1.08 | + 0.42 | 98.0 | 7,140 | 1.91 | " |
| | 3 | + 0.48 | + 0.42 | 100.5 | 6,310 | 1.53 | " |
| 2-1/2% Sodium Hypochlorite | 1 | - 0.46 | nil | 96.5 | 7,840 | 2.14 | " |
| | 2 | - 0.36 | + 0.43 | 97.0 | 7,850 | 2.56 | " |
| | 3 | - 0.53 | + 0.43 | 95.5 | 6,820 | 2.14 | " |
| 5% Acetic Acid | 1 | nil | + 0.89 | 77.0 | 6,600 | 1.50 | " |
| | 2 | + 0.10 | + 0.42 | 74.5 | 6,450 | 1.48 | " |
| | 3 | - 0.39 | + 0.89 | 73.0 | 7,040 | 1.38 | " |
| Ethyl Acetate | 1 | + 4.03 | + 1.28 | 106.0 | 4,140 | 2.15 | " |
| | 2 | + 3.82 | + 1.27 | 98.0 | 5,250 | 2.86 | " |
| | 3 | + 3.92 | + 1.27 | 104.5 | 3,705 | 2.87 | " |
| 2% Sodium Hydroxide | 1 | + 5.29 | + 1.27 | 86.5 | 6,700 | 2.22 | " |
| | 2 | + 5.30 | + 0.85 | 89.0 | 6,710 | 1.93 | " |
| | 3 | + 5.10 | + 2.14 | 89.0 | 6,470 | 1.95 | " |
| 2% Ammonium Hydroxide | 1 | + 4.09 | + 0.85 | 99.0 | 7,020 | 3.17 | " |
| | 2 | + 3.98 | + 0.85 | 100.0 | 7,190 | 3.13 | " |
| | 3 | + 4.04 | + 1.28 | 99.0 | 7,310 | 2.66 | " |
| Kerosene | 1 | + 0.70 | nil | 103.5 | 8,190 | 3.20 | " |
| | 2 | + 0.67 | nil | 104.0 | 7,640 | 3.25 | " |
| | 3 | + 0.70 | + 0.43 | 102.5 | 8,530 | 2.60 | " |
| Methyl Isobutyl Ketone | 1 | + 1.29 | nil | 101.0 | 6,830 | 3.26 | " |
| | 2 | + 1.74 | nil | 102.5 | 7,300 | 2.57 | " |
| | 3 | + 0.59 | nil | 105.0 | 9,170 | 2.46 | " |
| Ethylene Dichloride | 1 | + 4.88 | + 1.28 | 103.0 | 3,920 | 2.78 | " |
| | 2 | + 4.64 | + 0.43 | 106.5 | 5,340 | 2.88 | " |
| | 3 | + 4.61 | + 0.43 | 105.5 | 5,120 | 2.70 | " |

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## TABLE III

### CHEMICAL RESISTANCE OF UCNC 640-02

Test Conditions: Ambient Temperature; Static Conditions; 182 days

| Test Media | Spec. No. | % Wt. Chg. | % Dim. Chg. | ASTM D-785-51 Rockwell Hardness "R" Scale | ASTM D-790-58T Flexural Strength, PSI | ASTM D-790-58T Modulus in Flexure, PSI X $10^6$ | Remarks |
|---|---|---|---|---|---|---|---|
| Control | 1 | --- | --- | 109.0 | 6,190 | 2.14 | Unexposed |
| | 2 | --- | --- | 109.5 | 6,480 | 2.30 | " |
| | 3 | --- | --- | 107.0 | 8,000 | 2.83 | " |
| 2% Hydrochloric Acid | 1 | + 8.30 | + 0.40 | - 17.0 | 2,095 | 1.35 | Softened and Laminated |
| | 2 | +11.82 | - 1.18 | - 14.0 | 2,215 | 1.28 | |
| | 3 | + 7.08 | - 2.40 | - 16.0 | 2,688 | 1.44 | " |
| 2% Sulfuric Acid | 1 | +13.05 | + 8.39 | 15.0 | 2,880 | 1.54 | " |
| | 2 | +14.07 | + 8.72 | 12.5 | 2,365 | 1.08 | " |
| | 3 | + 7.57 | + 3.96 | 14.0 | 2,745 | 1.45 | " |
| 2% Nitric Acid | 1 | +16.10 | + 1.98 | 85.0 | 2,400 | 1.75 | " |
| | 2 | +16.95 | + 1.59 | 18.0 | 2,835 | 1.50 | " |
| | 3 | +15.63 | + 1.98 | - 1.0 | 2,645 | 1.56 | " |
| 2-1/2% Sodium Hypochlorite | 1 | +16.87 | nil | 85.0 | 3,680 | 2.06 | No apparent change |
| | 2 | +17.30 | nil | 76.0 | 3,750 | 1.97 | " |
| | 3 | +18.10 | nil | 65.0 | 2,535 | 1.48 | " |
| 5% Acetic Acid | 1 | + 9.93 | + 3.98 | 46.0 | 1,048 | 0.507 | Softened and Laminated |
| | 2 | + 9.36 | + 3.58 | 54.0 | 1,512 | 0.751 | |
| | 3 | +14.28 | + 1.60 | 87.0 | 1,812 | 1.54 | " |
| Ethyl Acetate | 1 | + 4.23 | nil | 75.0 | 2,665 | 1.77 | No apparent change |
| | 2 | + 5.08 | nil | 104.0 | 4,255 | 2.50 | " |
| | 3 | + 3.12 | nil | 102.0 | 4,855 | 2.53 | " |
| 2% Sodium Hydroxide | 1 | +10.85 | + 0.40 | 71.0 | 2,420 | 2.09 | " |
| | 2 | +16.50 | nil | 90.0 | 3,780 | 1.99 | " |
| | 3 | +14.50 | + 0.40 | 84.0 | 2,110 | 1.37 | " |
| 2% Ammonium Hydroxide | 1 | +21.82 | + 0.80 | 123.0 | 7,940 | 2.87 | " |
| | 2 | +25.80 | nil | 122.0 | 6,550 | 2.38 | " |
| | 3 | +24.95 | + 0.80 | 121.5 | 5,330 | 2.81 | " |
| Kerosene | 1 | +13.10 | + 0.80 | 106.0 | 5,710 | 2.86 | Saturated |
| | 2 | +13.65 | nil | 101.0 | 6,180 | 2.47 | " |
| | 3 | +12.67 | nil | 93.0 | 5,710 | 2.53 | " |
| Methyl Isobutyl Ketone | 1 | + 9.73 | nil | 101.0 | 4,895 | 2.56 | No apparent change |
| | 2 | + 8.69 | + 0.40 | 101.0 | 5,370 | 2.56 | " |
| | 3 | + 5.34 | nil | 102.0 | 5,570 | 2.85 | " |
| Ethylene Dichloride | 1 | +10.42 | + 0.40 | 96.0 | 4,750 | 2.46 | " |
| | 2 | + 8.06 | nil | 74.5 | 3,875 | 2.07 | " |
| | 3 | + 8.67 | - 0.40 | 97.0 | 5,480 | 2.56 | " |

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## TABLE IV

## CHEMICAL RESISTANCE OF UCNC 766-37

Test Conditions: Ambient Temperature; Static Conditions; 182 days

| Test Media | Spec. No. | % Wt. Chg. | % Dim. Chg. | ASTM D-785-51 Rockwell Hardness "M" Scale | ASTM D-790-58T Flexural Strength, PSI | ASTM D-790-58T Modulus in Flexure, PSI X $10^6$ | Remarks |
|---|---|---|---|---|---|---|---|
| Control | 1 | --- | --- | 103.5 | 11,600 | 1.98 | Unexposed |
| | 2 | --- | --- | 103.0 | 12,625 | 2.08 | " |
| | 3 | --- | --- | 105.5 | 10,100 | 2.18 | " |
| 2% Hydrochloric Acid | 1 | - 3.08 | + 0.84 | 98.0 | 10,525 | 1.30 | Laminated |
| | 2 | - 2.66 | + 0.42 | 101.0 | 10,950 | 1.17 | " |
| | 3 | - 3.13 | + 0.42 | 101.5 | 10,400 | 1.48 | " |
| 2% Sulfuric Acid | 1(1) | --- | --- | --- | --- | --- | --- |
| | 2 | - 4.08 | + 1.27 | 98.5 | 9,860 | 1.53 | Laminated |
| | 3 | - 4.29 | + 0.42 | 100.5 | 9,640 | 1.37 | " |
| 2% Nitric Acid | 1 | - 8.22 | - 0.41 | 104.5 | 10,675 | 1.41 | Slight lamination |
| | 2 | - 2.55 | nil | 104.0 | 11,650 | 1.94 | " |
| | 3 | - 5.37 | + 0.41 | 103.0 | 11,660 | 1.70 | " |
| 2-1/2% Sodium Hypochlorite | 1 | - 0.11 | nil | 100.0 | 10,475 | 1.99 | Slight discoloration |
| | 2 | + 0.51 | + 0.41 | 100.5 | 9,800 | 1.74 | |
| | 3 | + 0.34 | nil | 100.5 | 9,320 | 1.96 | " |
| 5% Acetic Acid | 1 | + 1.17 | + 0.41 | 101.5 | 9,300 | 1.46 | No apparent change |
| | 2 | + 1.06 | + 0.84 | 102.0 | 11,225 | 1.64 | " |
| | 3 | + 1.29 | + 0.83 | 100.5 | 10,775 | 1.58 | " |
| Ethyl Acetate | 1 | - 0.33 | - 0.41 | 101.0 | 10,500 | 1.96 | " |
| | 2 | - 0.34 | nil | 103.0 | 11,430 | 2.06 | " |
| | 3 | - 0.38 | nil | 100.0 | 10,560 | 2.02 | " |
| 2% Sodium Hydroxide | 1 | + 4.64 | + 1.67 | 95.0 | 9,190 | 1.66 | Adherent film |
| | 2 | + 4.16 | + 1.68 | 93.5 | 10,325 | 1.54 | " |
| | 3 | + 5.58 | + 1.24 | 92.5 | 8,470 | 1.43 | " |
| 2% Ammonium Hydroxide | 1 | + 3.63 | + 0.83 | 105.0 | 8,130 | 2.26 | No apparent change |
| | 2 | + 3.60 | + 0.41 | 105.5 | 9,620 | 2.40 | " |
| | 3 | + 3.61 | + 0.83 | 105.0 | 9,950 | 2.39 | " |
| Kerosene | 1 | - 0.14 | - 0.41 | 104.5 | 9,900 | 2.53 | " |
| | 2 | - 0.16 | - 0.82 | 102.0 | 10,425 | 2.48 | " |
| | 3 | - 0.16 | nil | 106.5 | 10,625 | 2.00 | " |
| Methyl Isobutyl Ketone | 1 | - 0.43 | - 0.41 | 102.5 | 11,775 | 1.99 | " |
| | 2 | - 0.50 | nil | 103.0 | 11,220 | 2.02 | " |
| | 3 | - 0.43 | - 0.41 | 103.0 | 10,900 | 2.03 | " |
| Ethylene Dichloride | 1 | - 0.29 | + 1.25 | 105.5 | 10,150 | 2.42 | " |
| | 2 | - 0.41 | nil | 103.5 | 12,500 | 2.32 | " |
| | 3 | - 0.27 | nil | 104.5 | 12,180 | 2.35 | " |

(1) Specimen used for earlier demonstration and photographs.

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TABLE V

APPRAISAL OF CHEMICAL RESISTANCE OF ASBESTOS MATERIALS

(Arbitrary limits of changes in mechanical properties are assigned as indicated in left column)(1)

Exposed: 182 days

| Test Media | 2% Hydrochloric Acid | 2% Sulfuric Acid | 2% Nitric Acid | 2-1/2% Sodium Hypochlorite | 5% Acetic Acid | Ethyl Acetate | 2% Sodium Hydroxide | 2% Ammonium Hydroxide | Kerosene | Methyl Isobutyl Ketone | Ethyle Dichlor |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **Transite** | | | | | | | | | | | |
| 5% Change in Weight | U | U | U | U | S | S | U | U | U | S | U |
| 5% Change in Dimensions | S | U | S | S | S | S | S | S | S | S | S |
| 30 Points Change in Hardness | U | U | S | S | U | S | S | S | S | S | S |
| 25% Loss in Strength | U | U | U | U | U | U | U | S | U | U | U |
| 25% Loss in Modulus | S | U | S | S | U | S | S | S | S | S | S |
| Disposition | U | U | U | U | U | U | U | S | U | U | U |
| **UCNC 465-15** | | | | | | | | | | | |
| 5% Change in Weight | S | S | S | S | S | S | U | S | S | S | S |
| 5% Change in Dimensions | S | S | S | S | S | S | S | S | S | S | S |
| 30 Points Change in Hardness | S | S | S | S | S | S | S | S | S | S | S |
| 25% Loss in Strength | U | U | U | S | U | U | U | U | S | S | U |
| 25% Loss in Modulus | U | U | S | S | U | S | S | S | S | S | U |
| Disposition | U | U | U | S | U | U | U | U | S | S | U |
| **UCNC C40-02** | | | | | | | | | | | |
| 5% Change in Weight | U | U | U | U | U | S | U | U | U | U | U |
| 5% Change in Dimensions | S | U | S | S | S | S | S | S | S | S | S |
| 30 Points Change in Hardness | U | U | U | S | U | S | S | S | S | S | S |
| 25% Loss in Strength | U | U | U | U | U | U | U | S | S | S | U |
| 25% Loss in Modulus | U | U | U | S | U | S | S | S | S | S | S |
| Disposition | U | U | U | U | U | U | U | S | S | S | U |
| **UCNC 766-37** | | | | | | | | | | | |
| 5% Change in Weight | S | S | U | S | S | S | S | S | S | S | S |
| 5% Change in Dimensions | S | S | S | S | S | S | S | S | S | S | S |
| 30 Points Change in Hardness | S | S | S | S | S | S | S | S | S | S | S |
| 25% Loss in Strength | S | S | S | S | S | S | S | S | S | S | S |
| 25% Loss in Modulus | U | U | S | S | S | S | S | S | S | S | S |
| Disposition | S | S | S | S | S | S | S | S | S | S | S |

S = Satisfactory
U = Unsatisfactory

(1) VanDelinder, L. S., and Cook, D. C., Materials of Construction – Evaluation of Rigid Plastics for Organic Chemical Service, February 8, 1962 (ML:500:69-F1)

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UC-000 #80

SUMMARY REPORT

# PLASTICS INTERMEDIATES
# POLYESTER RESINS
# HIGH PURITY ASBESTOS AS A THIXOTROPE FOR POLYESTERS

**Authors:** C. W. McGary
P. L. Smith

**Date:** March 18, 1965

**Project No.:** 119E21

**File No.:** 3780

SUMMARY High purity asbestos from the Mining and Metals Division can be used as a viscosity modifier and thixotropic aid in polyesters. A concentration of 2.5-3.0 per cent asbestos in a hand lay-up polyester provides thixotropy equivalent to 1.0 per cent Cab-o-sil.

INTRODUCTION Polyester resins (40 per cent styrene) for hand lay-up and spray-up applications customarily are modified by addition of 1.0 per cent Cab-o-sil. Reportedly,(1) viscosity of the resulting polyester, measured using a #2 spindle and a model LVT Brookfield viscometer, is as follows:

| Rpm | Viscosity range, cps |
|---|---|
| 6 | 900-1300 |
| 12 | 750-900 |
| 30 | 600-750 |
| 60 | 400-500 |

The purpose of this study was to determine whether high purity asbestos (6-8¢/lb.) could be used as a replacement for Cab-o-sil (72-74¢/lb.).

DISCUSSION The polyester used in this study was prepared using 2.0 moles of phthalic anhydride per 1.0 mole of maleic anhydride and a 10 per cent excess of propylene glycol. Viscosity, after dilution to 40 per cent styrene, was 275 cpoise (ref: 149PLS118).

Table I shows the effect of high purity asbestos and Cab-o-sil on viscosity of the polyester at 25°C. It is apparent that between 2.5 and 3.0 per cent asbestos provides thixotropy equivalent to 1.0 per cent Cab-o-sil. All of the samples showed signs of filler settling upon standing 1-2 hours at room temperature. Settling rate of asbestos was slower as the concentration increased.

Research and Development Department
Chemicals Division
Union Carbide Corporation

High purity asbestos, like Cab-o-sil, did not have an adverse effect on shelf life. Paraplex P-43 containing 3.0 per cent asbestos and 0.25 per cent cobalt naphthenate (6% cobalt) has been stored for 100 days at room temperature with no signs of gelation. (Ref: 149PLS72 and 88).

EXPERIMENTAL In order to establish equilibrium conditions rapidly, it was necessary to agitate the polyester-thixotrope mixture vigorously with a mechanical (Lightnin) mixer. Around 60 minutes of agitation was necessary for Cab-o-sil while 30 minutes was sufficient for the asbestos. The procedure used was as follows:

To the polyester was added 0.25 per cent cobalt naphthenate (6% cobalt) and the required amount of thixotrope. After bringing to 50°-55°C., the mixture was vigorously agitated without further heating. Weight of the mixture then was adjusted by addition of styrene to compensate for loss due to volatization and deaeration was effected in a vacuum dissicator. Viscosity at 25°C. then was determined using a #2 spindle (Model LVT Brookfield viscometer) at 6, 12, 30 and 60 rpm.

BIBLIOGRAPHY

(1) Oral communication to C. W. McGary from Charles Ross, Freeman Chemical Corp.

ATTACHMENTS: 1 Table

P. L. Smith

my

TABLE I

COMPARISON OF EFFECT OF HIGH PURITY ASBESTOS AND CAB-O-SIL ON VISCOSITY OF A HAND LAY-UP POLYESTER (40% STYRENE)

| | Thixotrope | | | |
|---|---|---|---|---|
| | HP Asbestos | | | Cab-o-sil |
| | 2% | 2.5% | 3.0% | 1.0% |
| Viscosity[1] @ 25°C., cps @ | | | | |
| 6 rpm | 725 | 900 | 1350 | 1150 |
| 12 rpm | 663 | 813 | 1225 | 913 |
| 30 rpm | 588 | 692 | >1000 | 665 |
| 60 rpm | >500 | >500 | >500 | >500 |

Ref: 149PLS124 and 125

(1) Using a #2 spindle and a model LVT Brookfield viscometer.

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UC 0001 F91

SUMMARY REPORT

# ISOPHORONE CATALYST DEVELOPMENT

ORIGINAL COPY

## EVALUATION OF ASBESTOS FOR USE AS ISOPHORONE CATALYST

**Authors:** A. L. Maloy
R. W. Halstead

**Date:** March 24, 1965

**Project No.:** 113A10

**File No.:** 3854

SUMMARY A catalyst prepared from asbestos has been evaluated in the laboratory reactor for use as an isophorone catalyst. The reaction stabilized at a moderately high conversion level with a chemical efficiency to useful products above 90 per cent. The ratio of mesityl oxide to isophorone, however, was approximately 3:1. This catalyst, therefore, would not be suitable for the production of isophorone. The catalyst does have merit for use to produce mesityl oxide.

INTRODUCTION Mr. J. B. Johnson, R and D business team representative, (No. 13) requested the evaluation of asbestos upon the recommendation of Mr. H. D. Kinsey (NYO). Had the asbestos resulted in a suitable catalyst, the economics of using it would be favorable because of the low cost and plentiful supply.

DISCUSSION The asbestos, which was received as a dry powder, was mixed with enough water to form a paste and was then extruded with a caulking gun into long cylindrical pieces approximately 3/8-inch in diameter. The material was dried and then broken into small sections about 1/2-inch long. The finished catalyst had a surface area of 53.5$M^2/g$ which is 2 to 5 times greater than the catalyst currently being used for the production of isophorone.

The laboratory reaction equipment used for this evaluation was described in Status Report 2915. The reaction parameters used for this investigation were as follows:

| | |
|---|---|
| Reactor Temperature, °C | 300-375 |
| System Pressure, psig | 30 |
| Catalyst Volume, ml | 1000 |
| Contact Time, Seconds | 64 |
| Reactor Feed | Acetone |

A reaction temperature of 355°C was maintained for the first 187 hours of operation to observe the decline in catalyst activity with catalyst age. A plot of conversion versus hours

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of operation, shown on Graph 1 in the attachments, indicates that the decline in activity after 187 hours of operation was less than 10 per cent. Also shown on this graph is the production of mesityl oxide and isophorone expressed in per cent of acetone conversion. As can be seen from the graph, the ratio of mesityl oxide to isophorone was approximately 3:1 throughout the 187 hours of operation. After 90 hours of operation, the chemical efficiency to useful products was 90 per cent or better for the duration of the run.

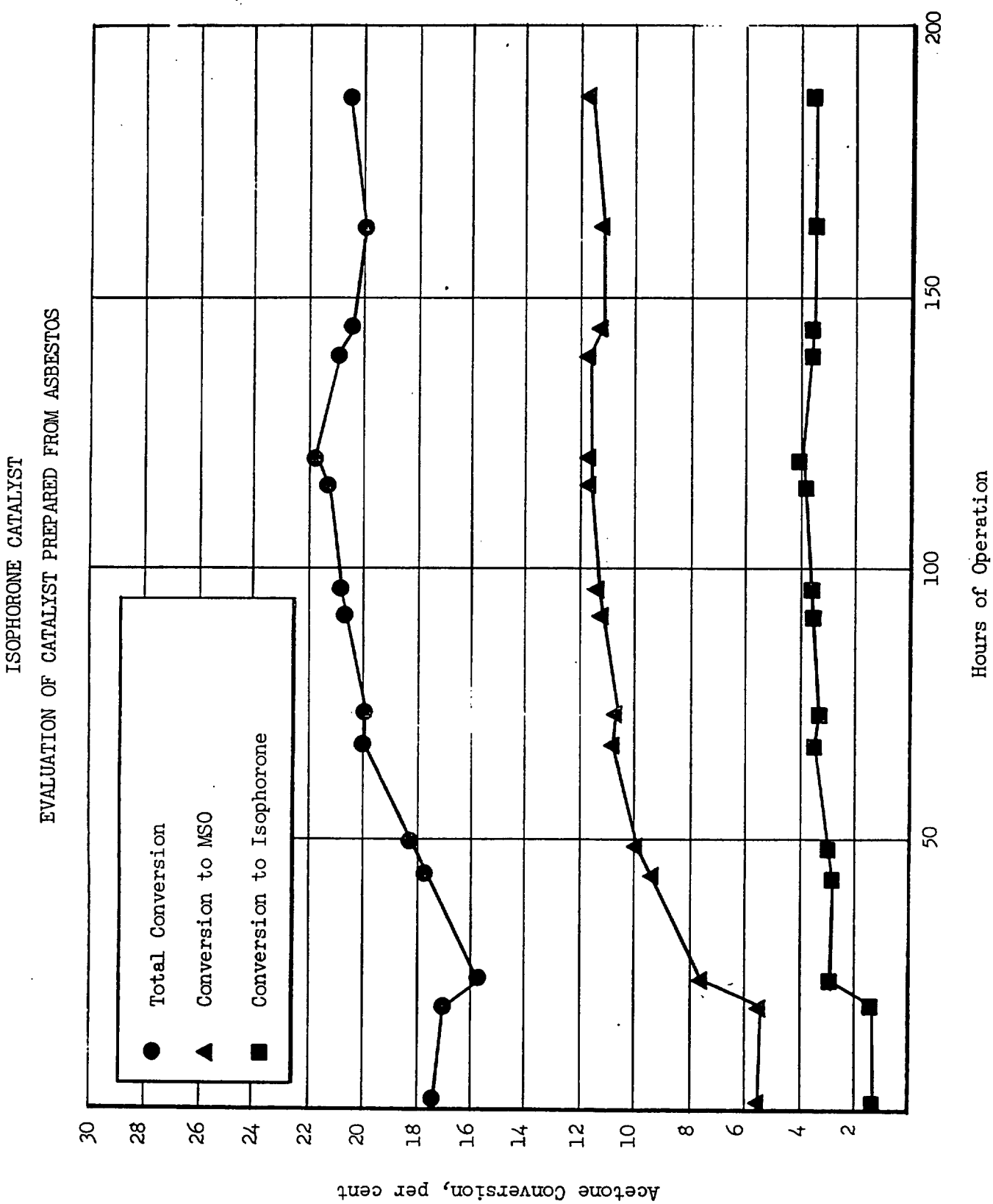
For the second phase of the investigation, the reaction was operated through a temperature range of 300-375°C to observe the influence of reaction temperature on catalyst performance. Graph 2, a plot of conversion versus reaction temperature, shows a significant increase in conversion when the temperature is increased from 300°C to 355°C. When the temperature was further increased to 375°C, the rate of increase in conversion was much lower. This can be attributed to the increased production of residues which deposited on the catalyst and masked the catalytic surface, thereby decreasing activity. The production of mesityl oxide and isophorone increased at about the same rate with increasing temperature. At reaction temperatures of 325°C and lower, the formation of residues was low and chemical efficiencies were in excess of 95 per cent.

CONCLUSIONS This material when considered for use as an isophorone catalyst, was quickly discredited because of the low conversion to the desired product. However, when operated at a reaction temperature of 300°C, asbestos shows considerable promise for use as a mesityl oxide catalyst. No attempt was made to thoroughly evaluate the catalyst for production of the latter material.

Attachments:
2 Figures

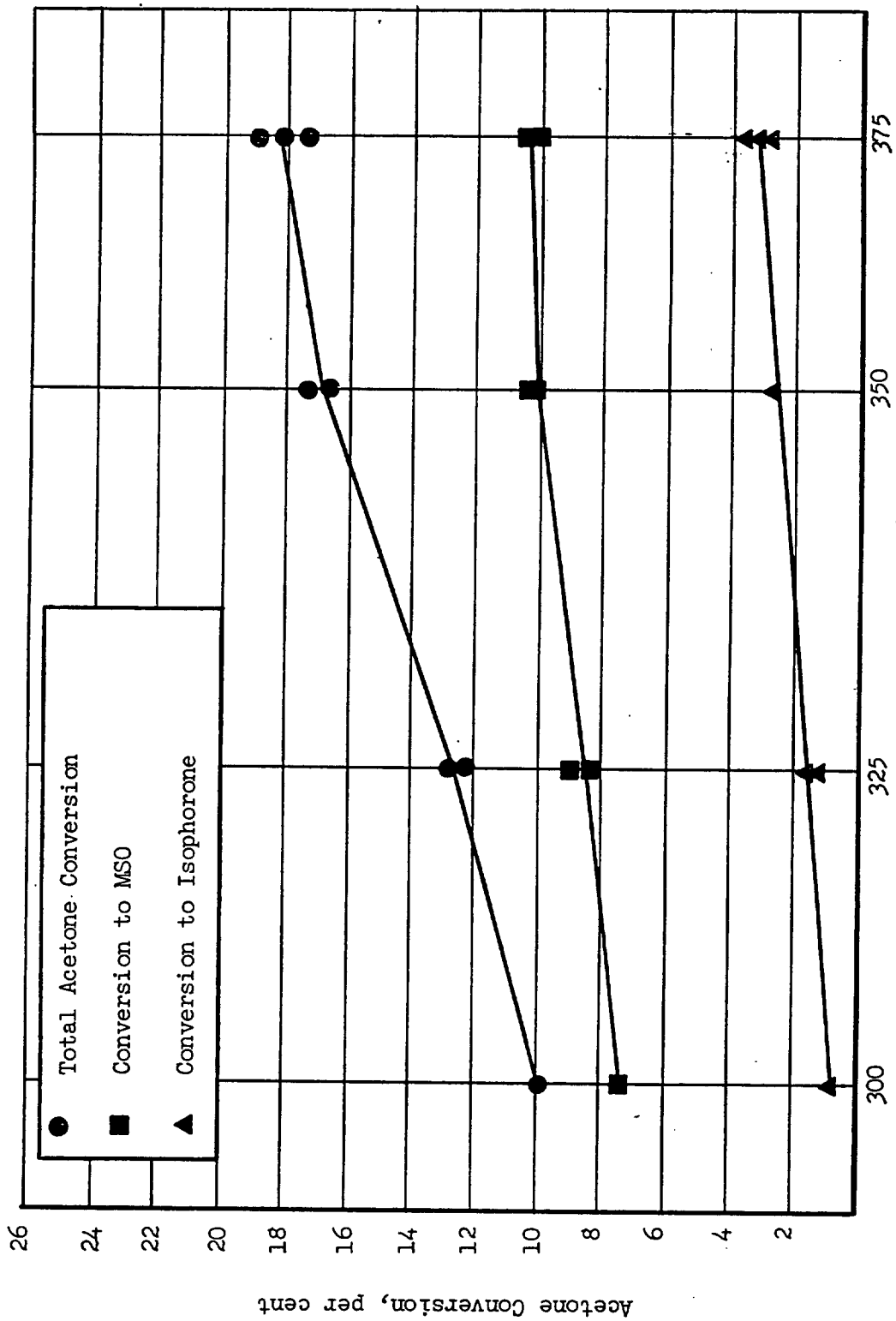
R.W. Halstead

whn


ISOPHORONE CATALYST
EVALUATION OF CATALYST PREPARED FROM ASBESTOS
Total Conversion
Conversion to MSO
Conversion to Isophorone
Acetone Conversion, per cent
Hours of Operation
30
28
26
24
22
20
18
16
14
12
10
8
6
4
2
50
100
150
200

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