INDUSTRIAL AND POWER CAPACITOR DEPARTMENT - GENERAL ELECTRIC COMPANY

Technical information for use by Monsanto Chemical Limited of United Kingdom in License Agreement on Chlorinated Diphenyl with Epoxide Additive - dated February 10, 1969.

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M.E. Scoville
February 10, 1969

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Industrial and Power Capacitor Department - General Electric Company

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1. Pyranol #1499 (#1242 Arochlor)	A13B1-S7
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M.E.Scoville
2/10/69

ENGINEERING
GENERAL

MEMO REPORT HF-68-808
April 8, 1968

COLUMN CHROMATOGRAPHIC REFINING OF PYRANOL II
PART I

J. E. Fagel
Material Physics and Chemistry Unit

ABSTRACT

Epoxide 206 and "aliphatic" concentration data are reported for Pyranol II chromatographic effluents from columns filled with fuller's earth (as received and thermally activated), silica gel, alumina, and magnesium silicate.

Probable explanations for typical Manufacturing experiences in the Pyranol II refining operation are derived from the data.

* * * *

Capacitor Department
Engineering Section
Advance Development Laboratory
Hudson Falls, New York

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INTRODUCTION

The refining of Pyranol II to yield a dielectric of acceptable resistivity and power factor is complicated by the Department's commitment to tank treating. Economics dictate recycling the large excesses of used Pyranol II following each treating operation, but a difficulty exists: refining methods successful in removing power dissipating impurities from the liquid also remove epoxide 206. Presaturation of the refining system with epoxide 206 is therefore necessary to permit Pyranol II purification without unacceptable, concomitant loss of epoxide. Fortunately, it appears possible to do this (remove power absorbers from PII without gross loss of epoxide); but, unfortunately, not without complication(s) resulting in part, at least, from the mode of reaction between epoxide 206 and the refining adsorbent (fuller's earth)--- cf. Memo Report HF-66-835.

Purification of liquids by column chromatography is now commonplace, efficient, and economical in many industrial, and even home environments. The Capacitor Department, in essence, employs column chromatography in its Pyranol refining operations, but in a grossly inefficient manner. It is highly probable that modern column chromatographic techniques and equipment (Hey, Culligan Man!) would substantially improve the Department's Pyranol refining by yielding higher quality oil with less fuss and bother, and without the "aliphatics" contamination that often characterizes the current Sparkler press operation. (No particular claim to originality is made for this statement; it seems to be well known and appreciated throughout the Department.)

The aim of A.D.L. work, the first part of which is here reported, is to point the way to a column procedure that can be introduced to the Department's Pyranol refining operation in place of the present system.

Further work will detail the results of column chromatographic purification of Pyranol and Pyranol II with respect to electrical characteristics, and the attempt to find correlation between such characteristics and chemical composition. Emphasis, however, will be on defining a practicable method for efficiently producing high quality Pyranol II both from virgin materials and from recycled oil.

EXPERIMENTAL

The chromatographic work was done with commercial glass columns 25 mm. in diameter. The columns were fitted with a simple constant-head device (water cooler principle) constructed from a conical funnel, and a separatory funnel as a reservoir. Desired flow rates were achieved by adjusting the height of the collection vessel (flexible, Teflon tubing connected to exit valve at column base). In each case, column beds contained 50 grams of adsorbent, 60-80 mesh; bed heights averaged about 20 cm. Flow rates were nominally about one ml per minute (extremes of 0.3 and 2 ml per minute). The constant head was approximately one meter.

The chromatographic adsorbents used were:

1. fuller's earth; as received in Capacitor Department
2. fuller's earth; thermally activated at 450°C, stored in air at 170°C
3. silica gel; 60-80 mesh, MC&B #9311
4. alumina; 60-80 mesh, Alcoa Type F-1, MC&B #9288
5. alumina; 60-80 mesh, Alcoa Type H-151, MC&B #9293
6. magnesium silicate (Florisil); 60-100 mesh, MC&B #9109

The two aluminas are essentially alike except H-151 has greater adsorptive capacity than F-1 (F-1 is normally used for gases; H-151 for liquids).

Epoxide and aliphatic concentrations were determined by conventional IR spectrography (Test Report HF-64-962).

Pyranol II (about 0.3 wt/wt percent) was synthesized by mixing one gallon quantities of refined storage Pyranol and epoxide 206 (vinylcyclohexene dioxide).

All column chromatography was performed at room temperature (19°C to 28°C - average close to 25°C). Column flow was interrupted overnight for all runs:- partly because continuous fraction collection equipment was not available, and partly because it was desirable to learn the effects, if any, of such intermittant operation. In normal use, a column would be operated more nearly continuously (there is normally no interest in collecting effluent fractions). Single pass operation was exclusive.

Data compilations, concentration calculations, integrated column retention value calculations, and plotting of column elution curves were all simultaneously performed via time-sharing computer.

RESULTS

The data are presented graphically in the accompanying elution curves. Effluent concentrations, expressed as fractions of starting (influent) concentrations, are plotted against effluent volume (25 ml fractions). The printed numbers represent grams of material retained by the column (integrated for each set of five effluent fractions).

The BASIC computer program XYPABS, appended, is a marriage of the library program XYPLOT and necessary additions to perform IR calibration conversions, integrated column retention calculations, and printout.

CONCLUSIONS AND DISCUSSION

A number of conclusions and projections are possible from this work, and the earlier work cited above (Memo Report HF-64-835).

From the earlier work it is evident that the principal fate of epoxide 206, on retention by fuller's earth, is polymerization. The exact mechanism of the polymerization reaction is not known, but presumably is closely related to documented heterogeneous polymerizations catalyzed on the surfaces of many metal oxides and similar solids: (e.g., Encyclopedia of Polymer Science and Technology, Interscience, Vol. 6, p.110ff., 1967). Runaway, violent polymerization does not occur except when critical quantities of fuller's earth and undiluted epoxide 206 are mixed; in particular, violence erupts when epoxide 206 is added to a slurry of epoxide 206 and fuller's earth - for reasons that are not immediately clear, but in attention - demanding fashion. In any event, epoxide 206 seems to be irreversibly adsorbed by fuller's earth even in non-violent reactions.

Historically, there has been considerable Department interest in the presence of aliphatic hydrocarbons in Pyranol. The total aromatic character of 1499 Pyranol makes the infrared spectrophotometric detection of impurity aliphatics (of which vacuum pump oil is one example) a simple task, and the determination of relatively small amounts of such impurities is routinely and sensitively performed. Epoxide 206 contains both active epoxide rings and aliphatic hydrocarbon functions in a single molecule (in addition, the roughly five percent impurities in commercial epoxide 206 bear further aliphatic functions). Therefore, "active epoxide" and "aliphatics" are synonymous in fresh Pyranol II (neglecting the five percent impurities in commercial epoxide 206). What, then, is the cause of increased aliphatics so often reported for Pyranol II? Pump oil is a probable "sometimes" answer, but obviously ridiculous as a general explanation. Polymerized epoxide 206 is the likely culprit.

Recently there has been interest in the relative merits of as received and of thermally activated fuller's earth. Reportedly, the use of as received material eliminates (or greatly lessens) the normal problem of high aliphatic content in refined Pyranol II. Why? The answer seems clear: activated earth would be expected to exert a greater catalytic effect on epoxide 206 polymerization than would unactivated earth, and hence would yield more polymer to contaminate the Pyranol II (or possibly polymer of readier solubility in Pyranol II). The hypothesis is this: polymerized epoxide 206 is generally retained by fuller's earth, but does exhibit some tendency to be stripped off by fresh Pyranol II. That is, polymerized epoxide 206, formed by reaction of Pyranol II with fuller's earth, is in small measure picked up by passage of subsequent Pyranol II over the earth on (in) which the polymer is largely retained. The polymerized epoxide 206 has lost none of the aliphatic character of the original monomer, but has lost all of its active epoxide character; hence the concentration of active epoxide is not changed in the Pyranol II, but the aliphatic hydrocarbon concentration is increased.

MONS 034787

Examination of the appended elution curves (I through VI) reveals the following:

1. The breakthrough for as received fuller's earth comes considerably sooner than for thermally activated earth. (Activation increases the adsorptive capacity of the earth.)

2. The breakthrough for the as received earth is sharp and clean - typical of physical adsorption without significant chemical reaction.

3. The breakthrough for the thermally activated earth is notably unsharp - characteristic of significant chemical change as well as physical adsorption.

4. After breakthrough, the active epoxide and aliphatic concentrations are essentially the same from as received fuller's earth columns (curves I & II, V & VI).

5. After breakthrough, active epoxide concentrations are notably less than the aliphatic concentrations from thermally activated fuller's earth columns (curves III & IV).

6. After long term elution (curves V & VI), there is a slight increase in aliphatic concentrations over active epoxide concentrations even for the as received fuller's earth. The difference is not at all comparable to that observed for the thermally activated earth; it is significant, however. In fact, the level of aliphatic is seen to exceed the epoxide (hence aliphatic) level in the influent - a fact that could result only from stripping the column of previously retained material.

The conclusion is that fuller's earth (particularly thermally activated fuller's earth), causes polymerization of epoxide 206 in Pyranol II; that the polymerized material is largely retained by the earth, but portions are subsequently stripped into fresh Pyranol II thereby increasing the aliphatic/epoxide ratio ("delta") of the latter.

Magnesium silicate is a compound similar to fuller's earth - the earth being a natural clay (a complex silicate of aluminum, magnesium, and potassium). Elution curves VII and VIII reveal the expected: Pyranol II responds to magnesium silicate in substantially identical fashion as to fuller's earth. (The magnesium silicate was used as received).

Epoxides are polymerized by acids and by various oxides. Silica gel is both acidic and an oxide; one would therefore predict a response of Pyranol II to silica gel much like that observed for activated fuller's earth. Elution curves IX and X, for as received silica gel, tend to confirm the prediction. Quantitative comparison is difficult at best for this, or in fact any other solid adsorbent system, because of uncertainties regarding the actual state of activation of the adsorbent. Thermally activated adsorbent is more active than non-activated material depending on time and temperature of the activation and on the length and quality of storage after activation. The significant and often unsuspected degradation of an activated solid during even brief storage under presumably good conditions was mentioned specifically in HF-64-835, and of course is generally well known. Undoubtedly many seemingly strange behaviors are readily accounted for by not-so-active "activated" adsorbents. Activated silica gel, as received from one commercial source, in any event, behaved much like Capacitor Department activated and stored fuller's earth.

Alcoa Type H-151 activated alumina, as received, exhibits an unusual and most interesting behavior in the column chromatography of Pyranol II (elution curves XI and XII). Breakthrough (allowing for interrupted column flow) is sharp indicating little if any chemical reaction - as one might predict for a nominally basic compound. Following the elution curves after breakthrough, one notes an unusual feature: the active epoxide effluent concentration tends to become greater than the aliphatic hydrocarbon concentration. It is easy to see how active epoxide can be lost through catalyzed polymerization, but how can the aliphatic function disappear without concomitant loss of active epoxide? The answer is that it can't; but the aliphatics included in the five percent of impurities in the commercial epoxide 206 (part of which are known to be acidic) can be adsorbed independently. Since the initial aliphatic concentration in Pyranol II synthesized from refined Pyranol (aliphatic-free) and commercial epoxide 206 includes both the aliphatic functions of the epoxide and indistinguishable (from the IR measurement viewpoint, at least) aliphatic contributions from the five percent impurities in the epoxide, the selective adsorption of the latter by an epoxide-"saturated" column would yield the unique result of an aliphatic/epoxide ratio less than one.

The basic alumina is expected to remove acidic compounds, whereas acidic adsorbents like silica gel, fuller's earth, etc., are not. The data of curves XI and XII are apparently the first non-electrical measure of the specific removal of impurities (more than likely power absorbing impurities, at least in part) from Pyranol II. Work in progress will hopefully confirm the electrical quality of such purified Pyranol II.

A precedent exists supporting in a small way the above claims. ES Dunham has recently shared with the writer essential details of work reported by Monsanto concerning their application of alumina-filled columns for chromatographic purification of 1499 Pyranol. Monsanto's experience (not in the least unusual or surprising in view of common chromatographic knowledge) has been that the alumina column purification has exhibited long term, high volume capacity and yielded dielectric of very high quality.

The cited elution curves for Pyranol II strongly suggest chromatographic purification with alumina should yield results comparable to Monsanto's for 1499. Aside from the general advantages of single-pass column operation over the recirculating Sparkler press procedure, the possibility of enjoying added gain by using alumina instead of fuller's earth is of increasing interest. Many engineering problems undoubtedly lie ahead, not to mention the inevitable economic factors that usually confuse otherwise good technical situations, but there may well be a small pot of gold worth seeking at the end of this rainbow.

The elution curves for all the adsorbents clearly reveal the effect of interrupted column flow. Weekend interruptions are discernible from overnight interruptions, even. The only immediate explanation for the sharp falloff of epoxide concentration coincident with each flow interruption is that given sufficient time (hours instead of minutes) of contact, the solids catalyze polymerization of significant amounts of epoxide. The fact that this behavior continues apparently ad infinitum indicates that too fast column flow is not the answer - in the indicated flow range, chromatographic equilibrium seems certain. In practice, then, Pyranol II could be refined by passage through an epoxide 206 saturated column without loss of epoxide as long as flow was continuous. Prolonged static contact of Pyranol II and any of the adsorbents should obviously be avoided.

The planned second part of this report will include electrical measurement data on column chromatographically purified Pyranol and Pyranol II, and will discuss specifics of column chromatography applicable to the Capacitor Department's refining operation.

ACKNOWLEDGMENT

The routine of column operations was handled mainly by E. Howk and V. Shedigian, both of whom also performed the many IR measurements.

Helpful discussions with many Department personnel (particularly including R.L. Guiles, G.A. Smith, E.S. Dunham and W.O. Solberg) are gratefully acknowledged.

```
XYPABS    9:56   TUESDAY  04-02-

0 REM ENTER DATA, LINE 150; LINES 160-299; CORRECT LINE 120
10 LET S=0
20 LET A=0
30 LET T=0
40 READ M,CO
50 GOTO 320
60 READ H
70 IF H= 999 THEN 790
80 LET A=A+1
90 LET S=S+H
100 LET Y= P/(M*CO)
110 IF A<>5 THEN 310
120 LET R= 85*1.39*(51-.00314-S/27400)
130 LET T=T+R
300 DATA 999
310 RETURN
320 PRINT
330 PRINT "WHAT ARE  YMIN,YMAX,XMIN,XMAX,DELX";
340 INPUT Y0,Y1,Y2,Y3,Y4
350 LET Y5=(Y1-Y0)/60
360 LET Y6=0
370 FOR Y = Y2 TO Y3 STEP Y4
380 GOSUB 60
390 IF Y6 = 0 THEN 650
400 IF Y6 = 20 THEN 430
410 PRINT " . ";
420 GOTO 450
430 PRINT " - ";
440 LET Y6 = 10
450 IF Y > Y1 THEN 620
460 IF Y < Y0 THEN 620
470 LET Y7 = Y0 + 2*Y5
480 LET Z=0740+5*Y5
490 IF Z<Y THEN 590
500 LET Y6 = Y6 + 1
510 IF Z-Y>=2*Y5 THEN 570
520 IF Z-Y>=Y5 THEN 550
530 PRINT "  +";
540 GOTO 710
550 PRINT " +";
560 GOTO 710
570 PRINT "+";
580 GOTO 710
590 LET Y7 = Y7 + 3*Y5
600 PRINT "   ";
610 GOTO 460
620 PRINT "USE SCALE:  (X,Y) =  "JXJ",  "JY
630 LET Y6 = Y6 + 1
640 GOTO 710
650 PRINT
660 PRINT "FOR Y:    TOP = "JY2J"  BOTTOM = "JY3J"  INCREMENT = "JY4
670 PRINT "FOR X:   LEFT = "JY0J"  RIGHT = "JY1J"  INCREMENT = "JY5
680 PRINT
690 PRINT "   I.........I.........I.........I.........I.........I.....▼
700 GOTO 430
710 IF A=5 THEN 740
720 PRINT
730 GOTO 770
740 PRINT R
750 LET A=0
760 LET S=0
770 NEXT Y
780 PRINT
790 PRINT "GRAND TOTAL RETAINED="JTJ"GRAMS"
800 END
```

MONS 034791

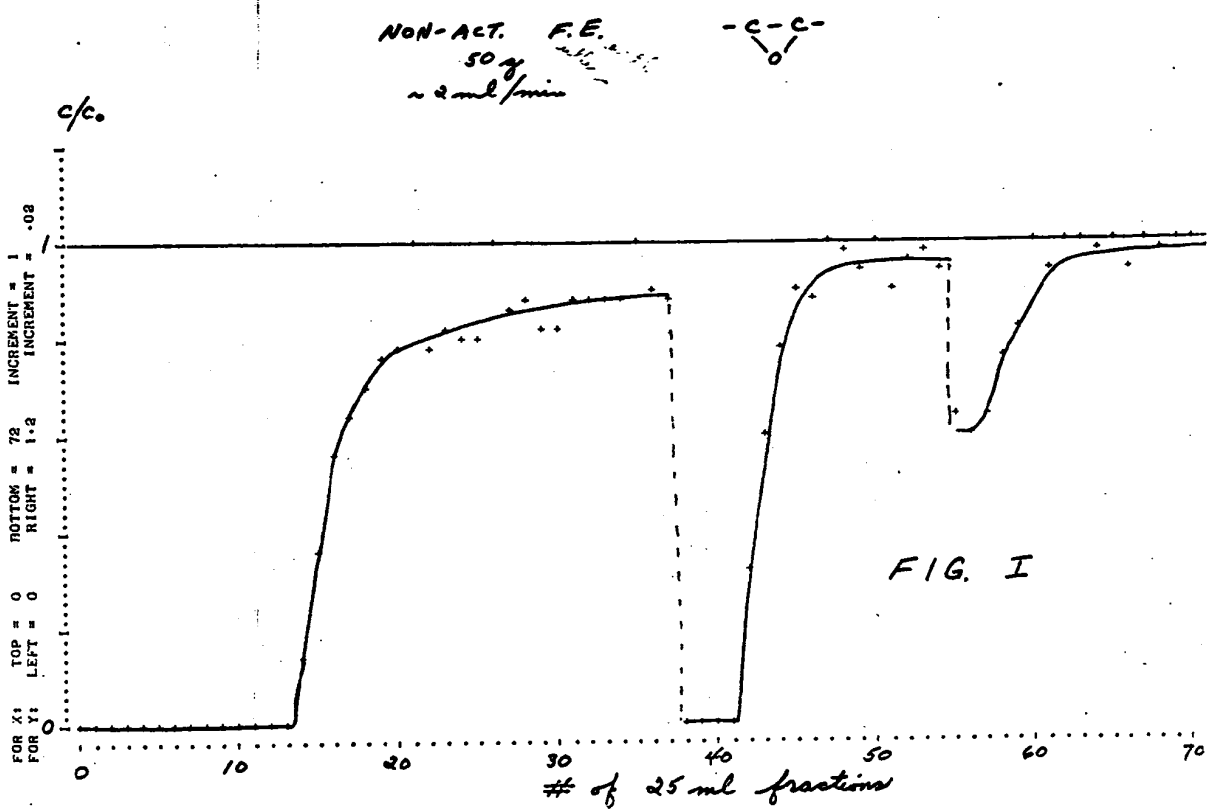
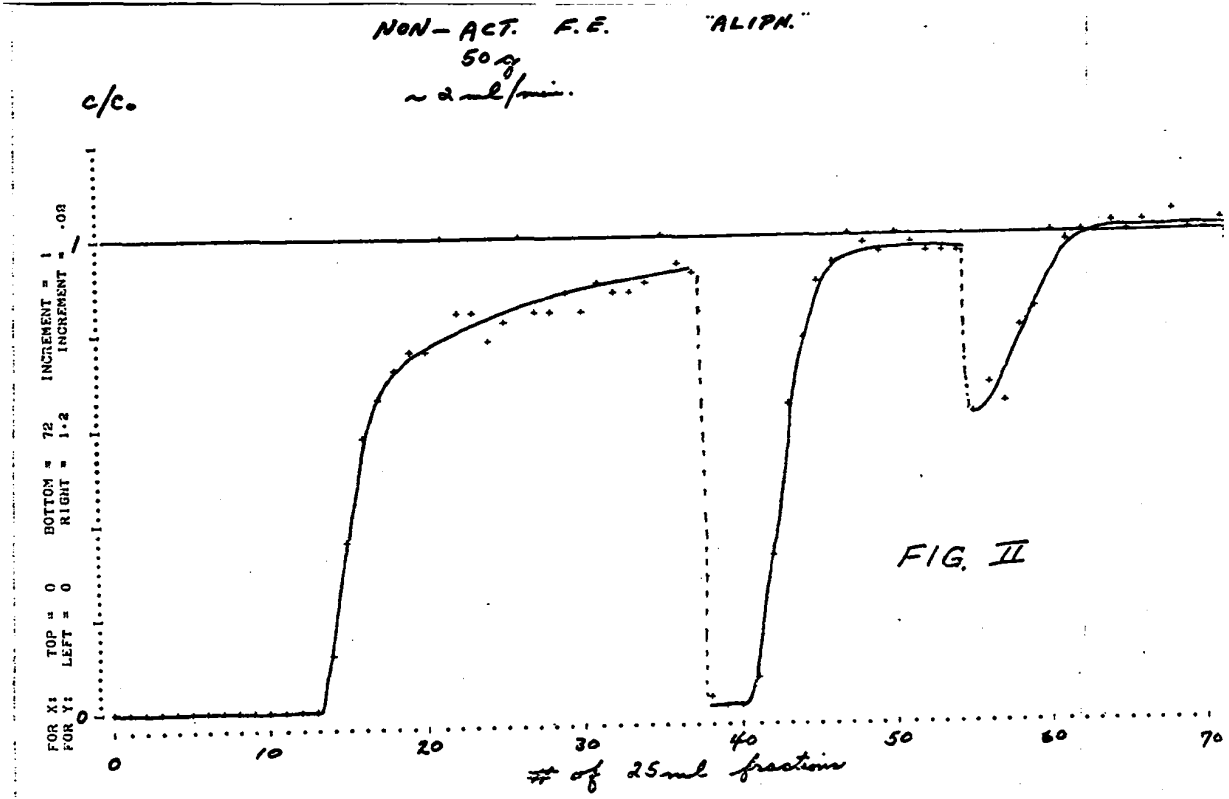

NON-ACT. F.E.
50 g
~ 2 ml/min
-C-C-
O
c/c.
1
0
FOR X: TOP = 0 BOTTOM = 72 INCREMENT = 1
FOR Y: LEFT = 0 RIGHT = 1.2 INCREMENT = .02
0
10
20
30
40
50
60
70
# of 25 ml fractions
MONS 034792

FIG. I


NON-ACT. F.E. "ALIPH."
50 g
~ 2 ml/min.
c/c.
FOR X: TOP = 0 BOTTOM = 72 INCREMENT = 1
FOR Y: LEFT = 0 RIGHT = 1.2 INCREMENT = .08
1
0
0
10
20
30
40
50
60
70
# of 25 ml fractions
FIG. II

MONS 034793

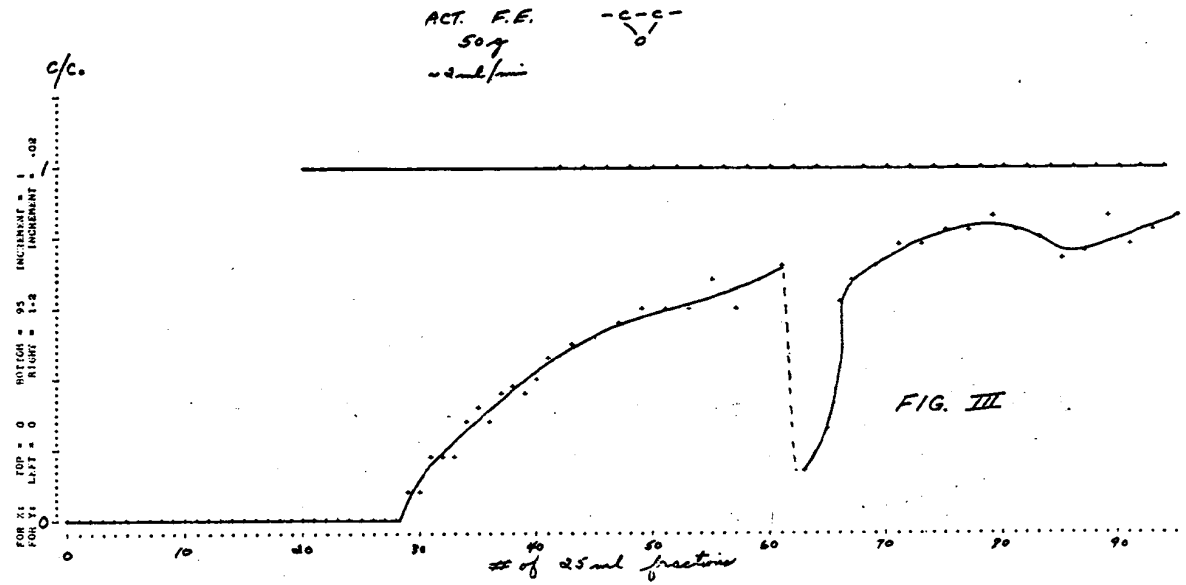


FIG. III

MONS 034794

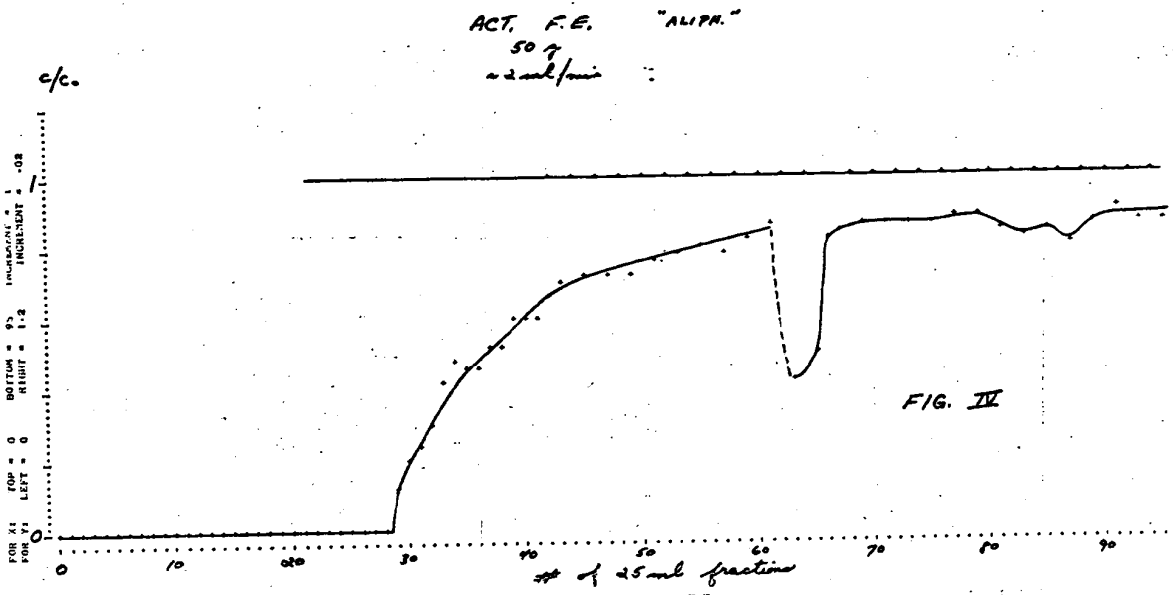


FIG. IV

MONS 034795

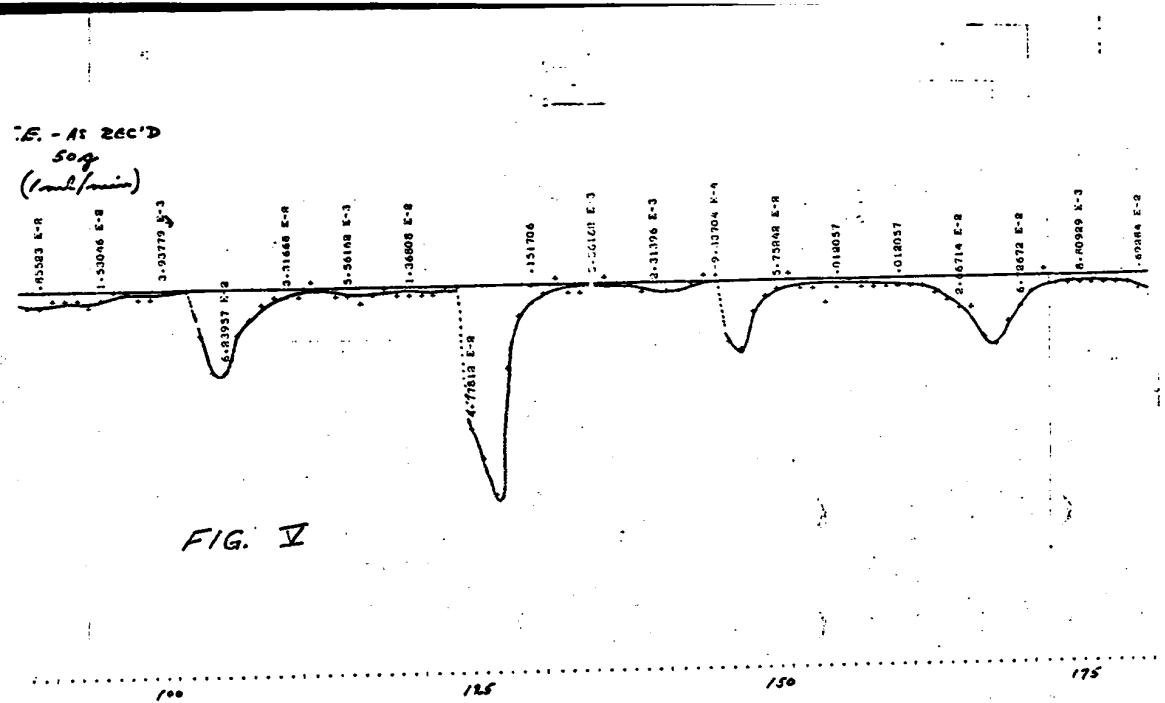
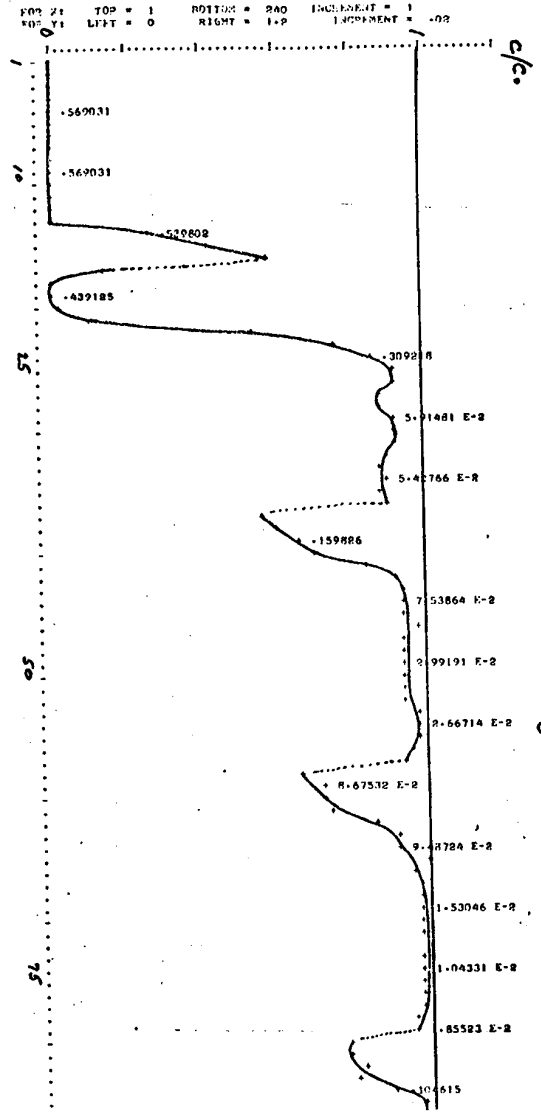


FIG. V

MONS 034796


FOR ZI TOP = 1 BOTTOM = 240 INCREMENT = 1
FOR YI LEFT = 0 RIGHT = 1+2 INCREMENT = .02
c/c.
.569031
.569031
.529802
.439185
.309216
5.91481 E-2
5.46766 E-2
.159886
7.53864 E-2
8.99191 E-2
8.66714 E-2
8.67532 E-2
9.48784 E-2
1.53046 E-2
1.04331 E-2
.85523 E-2
.104615
C-C

MONS 034797

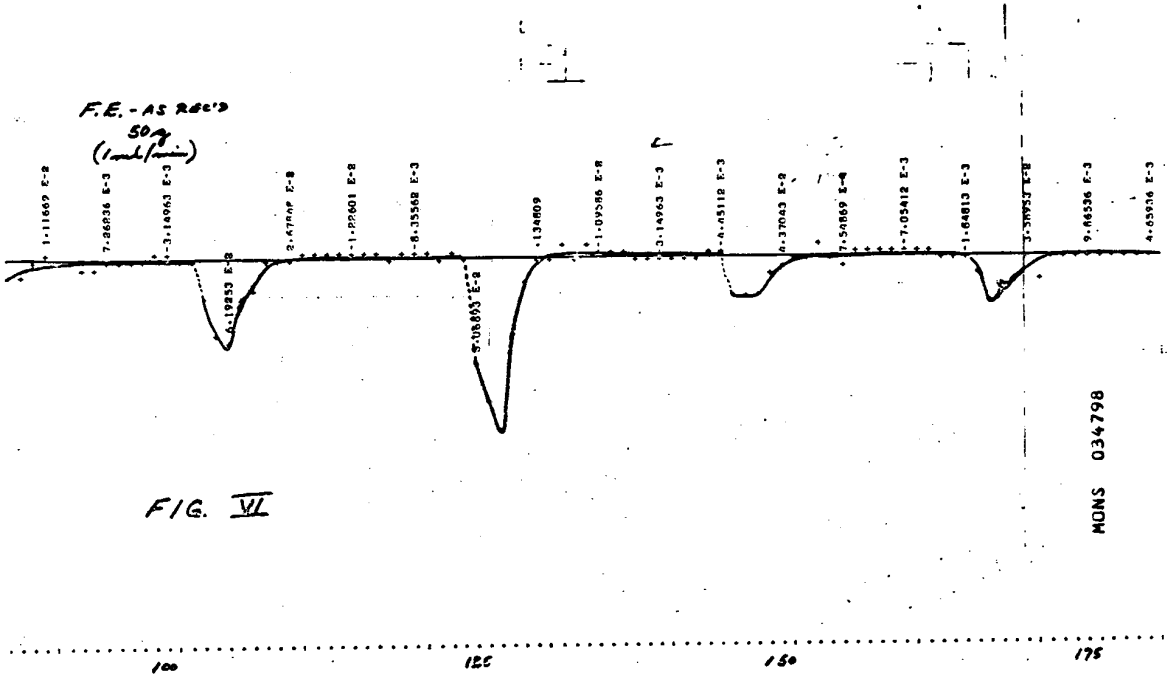
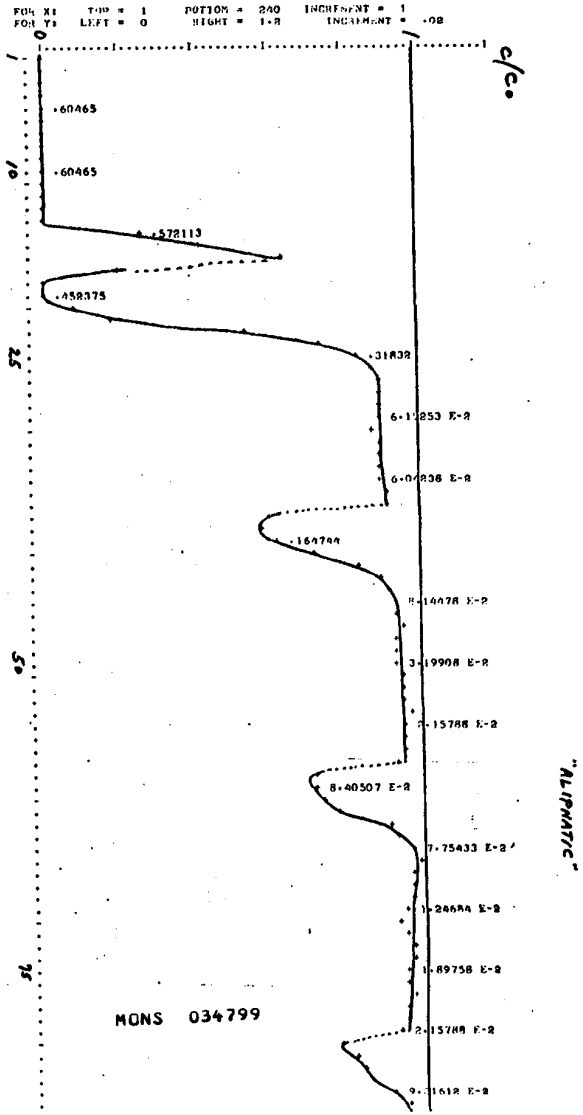


FIG. VI

MONS 034798


FOR X1 TOP = 1 POTTOM = 240 INCREMENT = 1
FOR Y1 LEFT = 0 RIGHT = 1.2 INCREMENT = .08
0
1
c/c.
.9
.8
.7
.6
.5
.4
.60465
.60465
.572113
.458375
.31838
6.12853 E-2
6.04838 E-2
.164799
8.14478 E-2
3.19908 E-2
2.15788 E-2
8.40507 E-2
7.75433 E-2
1.24684 E-2
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2.15788 E-2
9.31612 E-2
"ALIPHATIC"
MONS 034799

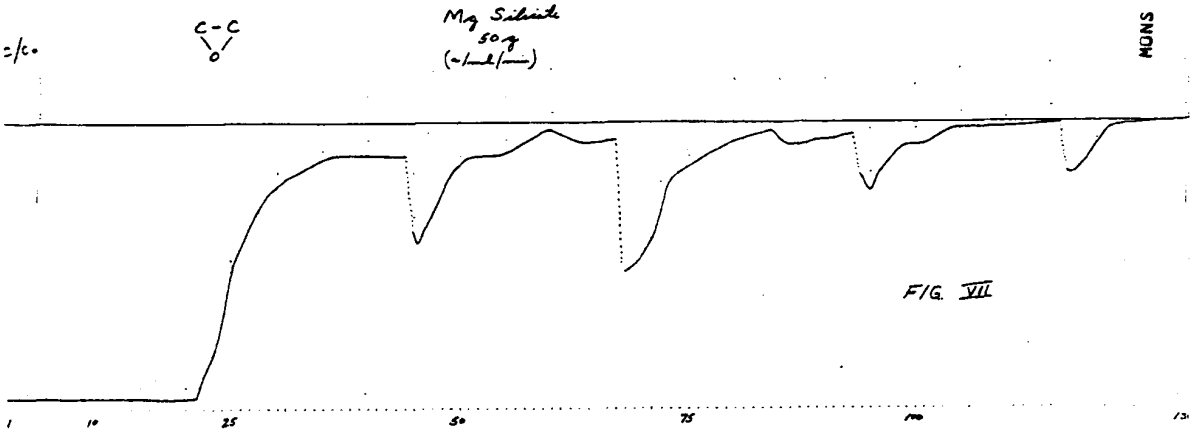


FIG. VII

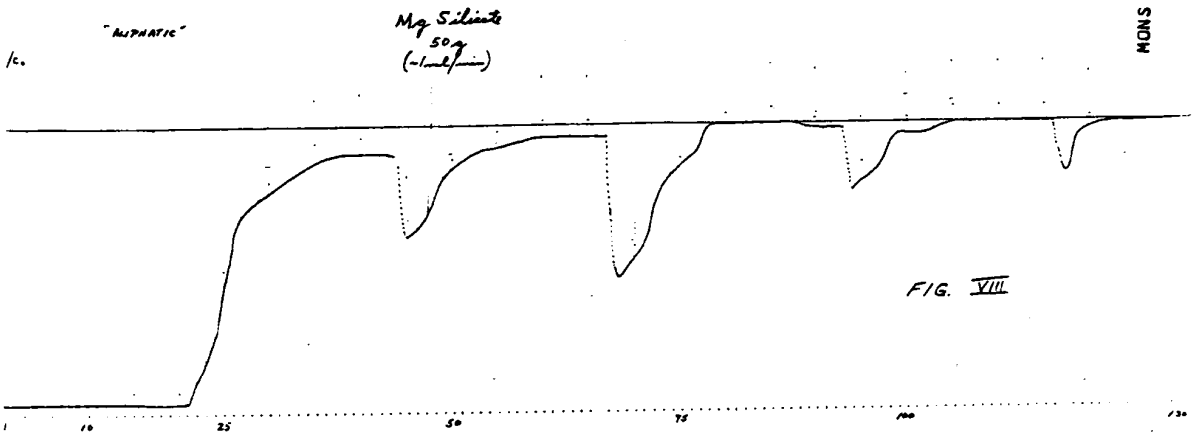


FIG. VIII

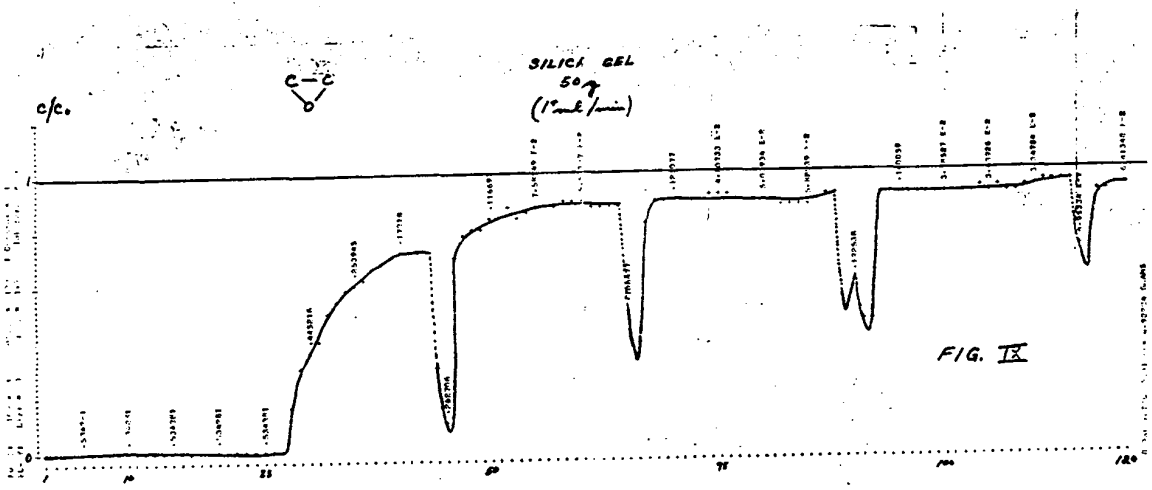


FIG. IX

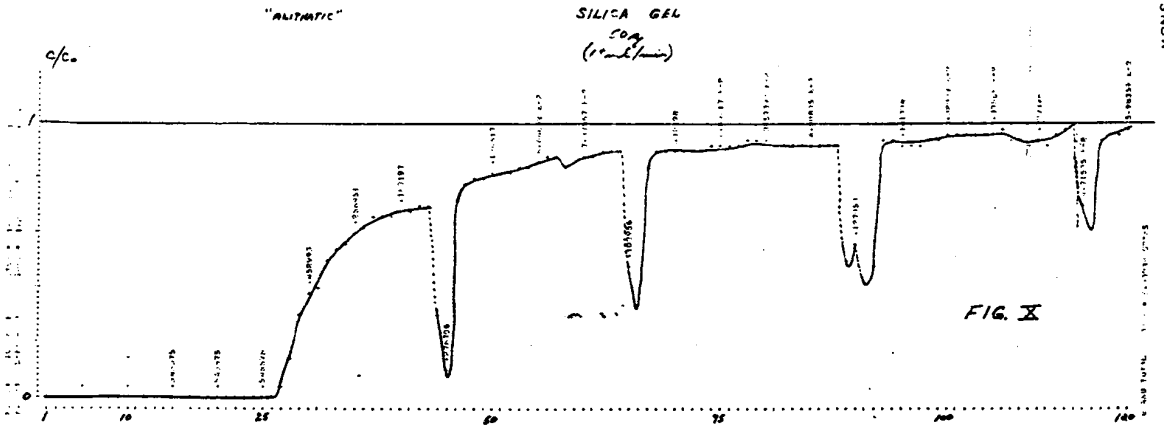
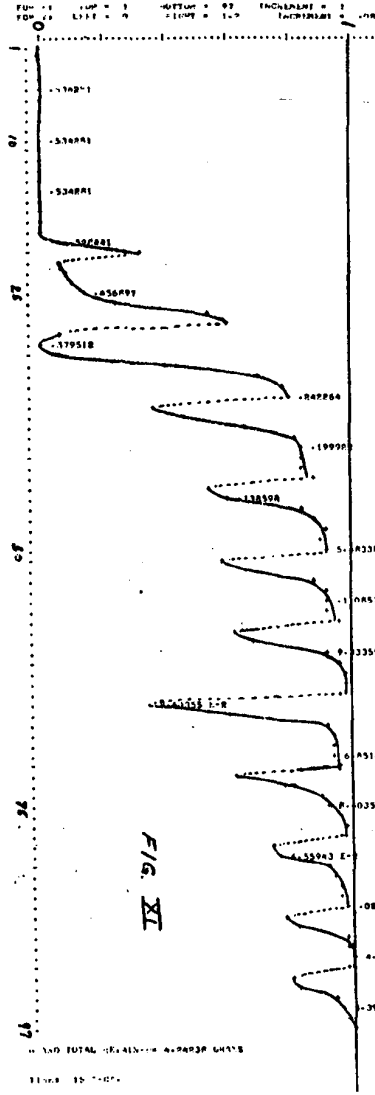


FIG. X



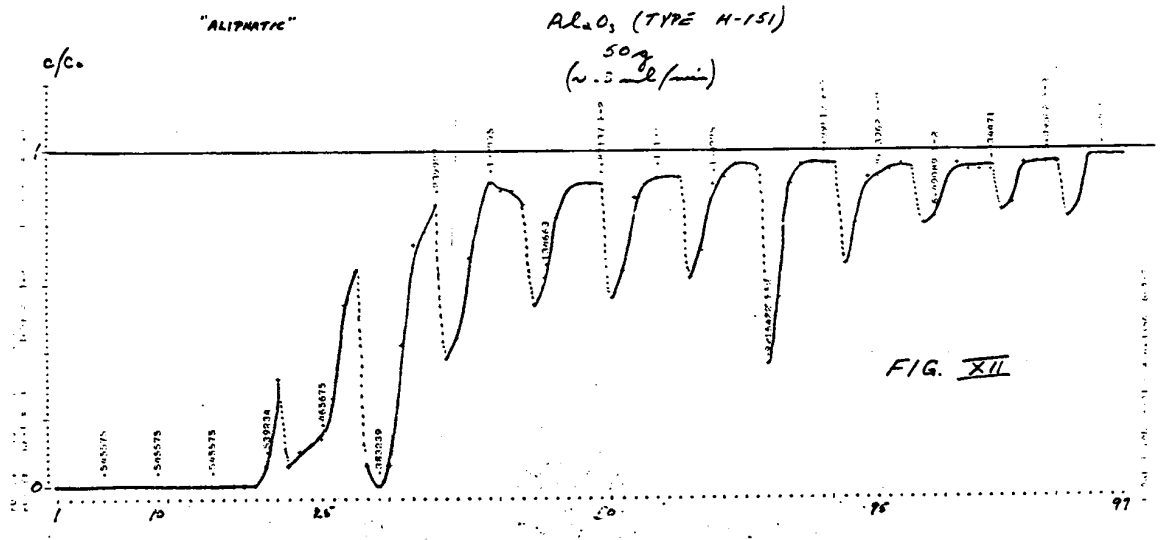


FIG. XII

MEMO REPORT HF-68-822
July 16, 1968

COLUMN CHROMATOGRAPHIC REFINING OF PYRANOL II. PART II.

J. E. Fagel
Material Physics and Chemistry

ABSTRACT

Single pass, room temperature column chromatographic purification of Pyranol II with both fuller's earth and alumina yields full strength PII with no "aliphatics", and 100°C-100 Hz dissipation of 0.6%. Minimum useful capacity is 4 liters per fifty grams of each adsorbent; breakthrough occurs at about 1 liter, and full strength effluent is obtained after 3 liters.

* * * * *

Capacitor Department
Engineering Section
Advance Development Laboratory
Hudson Falls, New York

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INTRODUCTION:

The results of initial column chromatographic efforts to purify Pyranol II were reported in Memo Report HF-68-808. Data in that report concerned column behavior, effluent concentrations of active epoxide and "aliphatics", holdup and breakthrough characteristics, but nothing about the electrical quality of the effluents. This report, and its companion describing the technique by which dissipation data were finally obtained, essentially completes the story started earlier. The conclusion, although not unexpected, is nonetheless happy.

EXPERIMENTAL:

Two columns, identical to those described in HF-68-808, were set up for the present work: The first filled with 50 g of 170°C air-dried fuller's earth, the second filled with 50 g of activated alumina H-151. Flow rates through each column (approximately 20 cm deep, each) were 0.3 ml/min under a constant head of roughly one meter. Percent dissipation at 100°C-100 Hz (and 1000 Hz), and epoxide and aliphatic concentration (IR) were determined for effluent samples from each column over a period of three weeks. Column flow was continuous throughout the entire period for each adsorbent (contrasted with the interrupted flow reported in HF-68-808).

RESULTS:

The data are tabulated in Tables I and II for each column.

DISCUSSION:

The % epoxide (column 3) values listed in the two tables were derived from IR absorption measurements of effluent samples taken from the Balsbaugh cell following dissipation measurement. Comparison samples measured before dissipation determinations showed full strength effluents after about 3000 ml. In other words, a roughly ten percent loss of active epoxide (no "aliphatic" change) as a consequence of the dissipation measurement is clearly revealed. The cause and/or mode of the loss are unknown; the loss, however, is as real as it is surprising, and raises a number of interesting questions regarding the actual in-service fate of epoxide 206 as a capacitor dielectric component. The immediate-interest significance of the loss seems minor, however; the loss certainly does not contradict earlier column chromatographic data.

The data further reveal effective purifying capacities of at least four-plus liters of Pyranol II per 50 grams of each adsorbent. The runs were terminated arbitrarily after the passage of roughly seven liters of PII; one might guess the actual capacities to be greater than the minimum value demonstrated, however.

The important, if not surprising, result of this demonstration is that a single, room temperature pass through modest-depth beds of either fuller's earth or alumina yields full strength Pyranol II of excellent 100°C-100 Hz dissipation. Q.E.D.!

What about the prognosis of the technique as a useful Department tool? The writer confidently expects the near-future implementation of the basic (columnar) approach by Manufacturing in some non-critical area, and gradually the complete replacement of existing refining facilities by a neat, efficient easily manageable, automated column operation.

Scaling columns to useful dimensions will of course uncover nuisance problems of the sort always encountered in the transition from laboratory through pilot plant to production. There is certainly no reason why anything greater than nuisance will be encountered, however, and at least a good trial installation should be constructable at modest cost and effort. The following specifics are suggested for openers:

1. A column roughly 12" in diameter and about 6' tall.

2. Fuller's earth activated at about 150°C (the usual higher temperature activation results only in unnecessary destruction of epoxide and the consequent generation of "aliphatics").

3. A simple pumping system for efficient liquid handling and pressurizing.

4. A simple heat exchanger to warm Pyranol to lower viscosity and hence speed column throughput (100°C is unnecessarily hot and possibly troublesome--increased undesired reaction rates--50°C to 75°C maximum is suggested).

5. Provision (pumping and storage) for recirculating liquid through column to facilitate initial "saturation" of adsorbent with epoxide.

6. At least 10X PII (that is, about 3% epoxide) for column "saturation." Probably considerably greater epoxide concentrations (10%-20%-----?) could profitably and safely be used, but continuous recognition must be given to certain consequences (all bad!) that result when "*too much*" epoxide is mixed with active adsorbent (Memo Report HF-64-835).

7. In line with No. 6, consideration should be given to the real possibility of making a concentrated "PII" that profitably could be added to refined 1499 to yield the desired final PII. It might be more economical to run the operation this way than conventionally (smaller volume of liquid through "PII" column, hence smaller column, etc, etc.). The concentrate concept warrants serious investigation, and reportedly (Monsanto personnel) is already employed in Europe.

As a first babystep, the writer has constructed an all glass column about 2" in diameter and 3.5' tall to produce usable quantities (laboratory scale, to be sure) of purified oil. An adsorbent charge of 500 grams of fuller's earth (160°C air dried) yields a bed about 17" deep, and a room temperature throughput of 300-plus ml per hour under a head of roughly four feet. This represents about a 15-fold output increase over the small (1" column, 50 g charge) setups originally used and described (Memo Report HF-68-808; first part of this report). The initial output from this column has a 100°C-100 Hz dissipation of 0.4%.

The data in Tables I and II offer further suggestion that alumina may be better suited to the PII purifying job than fuller's earth, and should be actively considered for future application. For the moment, however, there is no apparent reason not to proceed simply and directly using material on hand and well proven by long, direct experience. In other words, to summarize, the work reported and the suggestions offered say little more than this: A modest, reasonable change (to full column technique) of the present, basically proven procedure of Pyranol refining will yield a more efficient, reliable, and easily manageable system than presently available while permitting greater flexibility to cope with new problems the future will undoubtedly bring.

TABLE I

100°C - 100 Hz DISSIPTATION

FULLER'S EARTH COLUMN

Ml. Through Column	% D ‡	% Epoxide*		% Aliphatic*
380	.8	0		0
520	.7	0		0
905	.4	0		0
1300	.9	47		73
1450	.7	63		82
2445	.8	76		86
2965	.5	84)		95
3400	.7	87)		100
3875	.5	87)		95
4325	1.0	89)		100
5475	.6	92)	100 @	100
6305	.6	89)		100
6755	.6	89)		100
7205	.8	92)		100

‡ Influent %D = 2.5

* Relative to influent @ about 0.35% EP 206.

@ See discussion.

J. E. Engel
7-16-68

TABLE II

100°C - 100 Hz DISSIPTATION

ALUMINA COLUMN

Ml. Through Column	% D #	% Epoxide*		% Aliphatic*
325	.8	0		0
465	.7	0		0
815	.5	58		64
1275	.5	74		77
2355	.5	87		91
2875	.6	89		93
3325	.5	89	)	100
3785	.6	92	)	98
4235	.7	89	)	100
5315	.5	92	) 100 @	100
6215	.6	89	)	98
6665	.5	92	)	100
7115	.8	89	)	100

Influent %D = 2.5

* Relative to influent @ about 0.35% EP 206.

@ See discussion.

J. E. Fagel
7-16-68

MONS 034811

MEMO REPORT HF-68-821
July 16, 1968

TECHNIQUE FOR MEASURING DISSIPATION OF 1499 PYRANOL AND OF PYRANOL II

J. E. Fagel
Material Physics and Chemistry

ABSTRACT

Cell cleaning inadequacies are the principal source of contamination leading to the frequent experience of low precision dissipation measurements.

A technique based on this premise has been derived and found to yield reliable measurements routinely.

* * * *

Capacitor Department
Engineering Section
Advance Development Laboratory
Hudson Falls, New York

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INTRODUCTION:

The author in recent months has had need for reliable dissipation measurements, of relatively small volumes of Pyranol I and II, in support of column chromatographic purification studies. At the start of these studies, the naive assumption was made that the electrical measurements need could easily and routinely be met; [redacted]

In order to continue from the first part of the chromatographic work (Memo Report HP-68-808) it was necessary to develop a reliable dissipation measurement technique. Chemical and related measures of additive and of impurities concentrations in Pyranols are interesting and even pertinent, but the real proof of the pudding is of course electrical dissipation. The brief discussion offered in this memo relates a successful technique derived from the hypothesis that essentially the only reasonable cause for low quality dissipation measurements is contamination resulting from--or as residual of--cell cleaning inadequacies.

APPARATUS:

The Balsbaugh #3HV35 (Monel) dielectric liquid test cell was chosen because of minimal size (roughly 35 ml of liquid required), proven performance (Capacitor Department; ASTM), and immediate availability (two such cells have been located in the Department).

Instrumentation and associated equipment (completely shielded cell cage, for example) chosen was the GR Type 1605 Impedence Comparator setup in the QC Lab.--used with the generous approval and assistance of QC Lab. personnel.

TECHNIQUE:

The writer is of the opinion that reliable dissipation measurements must be possible given a clean test cell and (of course) functional, quality instrumentation. Likewise, measurements that fall short must suffer from contamination that in turn is directly attributable to inadequate cell cleaning. As previously indicated, the technique here described is based on the thesis of adequate cell cleaning, and basically consists of two parts: (1) Initial cleaning of cell, and (2) between measurements cleaning of cell.

1. Initial Cleaning

General experience, coupled with the dictates of the chemistry of the metal(s) of which typical cells are constructed, suggests an alkaline cleaning medium as most suitable for initial cleaning of the cell. (The Balsbaugh 3HV35 is Monel; 316 stainless steel would be preferred by the writer, and reportedly others - Monsanto personnel for example). Accordingly, the following is done: The disassembled cell is first cleaned with acetone to ensure removal of organic residues (principally Pyranol). The cell parts are then placed in a gently boiling aqueous solution (in a stainless steel beaker) containing roughly 10% (wt/vol) sodium

hydroxide and 1% ethylenediaminetetraacetic acid--the latter to stabilize cations and ensure that they stay in solution. Following boiling for approximately one-half hour, the parts are removed from the caustic solution, rinsed in water thoroughly, then rinsed in dilute, aqueous hydrochloric acid (1:10), and finally rinsed thoroughly again in distilled water, and oven dried at about 100°C.

2. Between Measurements Cleaning

Before the first measurement, and between each and every subsequent measurement, the entire (assembled) cell is cleaned with generous quantities of acetone and then hexane. The acetone is dispensed from a wash bottle in a fine stream to gain the benefit of motion in addition to solubility--this is an essential feature of the technique: Pyranol is not readily removed from the cell, it turns out. Cleaning is judged adequate when, (a) Pyranol is no longer precipitated by water from the acetone wash liquid, and (b) no residue is discernible on the cell surfaces (viewed obliquely from several different angles) following evaporation of the acetone. Repeated acetone washing is required to meet condition (a) and even further washing is essential to satisfy (b). Hot air (laboratory heat gun) is blown over the cell to prevent condensation of moisture from the air as a result of cooling caused by the evaporation of acetone.

Generous rinsing of all parts of the cell with hexane, and hot air drying, completes the cleaning procedure. The cell is then rinsed with two small portions (10 ml) of the sample to be measured , and finally filled with the sample.

The filled cell is placed in an oil bath, at about 105°C for 30 minutes. A thermal jacket (constructed with a glass beaker inside a larger, steel beaker--the annular space filled with glass wool) is kept in an oven at 100°C. The cell, on removal from the oil bath, is placed in the thermal jacket, and immediately transported to the comparator setup.

The completely closed cell cage precludes the convenient use of a controlled bath during measurement, but the initial, slight overheating of the cell, and the heated thermal jacket makes 100°C measurement quite simple and repeatable in the following manner: The dissipation at 100 Hz and at 1000 Hz is measured at the time in the cooling period of the cell when the observed capacitance equals the value initially measured for the cell at 100°C. This initial capacitance measurement was made at the time (about 5 minutes) after the removal of the cell from the oil bath when an enclosed thermocouple indicated a sample temperature of 100°C. The cell used for the measurements reported exhibited a capacitance of 187.7 pf, at 100°C, filled with either 1499 Pyranol or with Pyranol II. A duplicate cell has essentially the same capacitance. It is true that the actual temperature during measurement may not be exactly 100°C, but it is equally true that, (1) the temperature is close to 100°C (within a degree or two at most), and (2) it is repeatable whatever the exact value. Furthermore, it is repeatedly observed that the measured dissipation changes but little over the entire temperature range during the measurement; the absolute accuracy of the determined $\overline{D}$ is fully acceptable, in other words.

RESULTS:

Typical, replicated data obtained over a period of several days for a randomly selected sample of 1499 Pyranol (obviously of low quality) are tabulated in Table I along with calculated averages and standard deviations. Although the data are pooled, they actually come from two separate (and statistically distinct) populations: The first 13 values represent results obtained without washing the cell between measurements; the last 6 values were obtained following the previously described technique for cell cleaning. In other words, the 10% relative standard deviation is a worst-case expression of precision.

DISCUSSION:

One fundamental fault was discovered in the course of the work with the Balsbaugh cell: The large insulator (a filled polymer) separating the outer and guard electrodes of the cell was found to retain impurities to such an extent that its resistance dropped from 10^{12} ohms to 10^{5} ohms! No washing procedure was found that would successfully clean the insulator; but the high resistance could be restored by heating for at least one hour at 125°C in a vacuum oven. To circumvent the problem altogether, even though the vacuum-heat treatment was completely successful, new insulators were machined from Teflon--the same polymer being the insulation originally used between the inner and guard electrodes of the cell.

After prolonged use (three weeks; five measurements/day) and despite the fact nothing but AC ever was applied to the cell, an unmistakable yellow-brown film had formed on the walls of the cell, along with a few darker colored spots on the bottom of the cell. Both blemish conditions were easily removed by mild abrasion with sink cleaner (Bab-o; special institutional formula, to be exact). Initial cleaning procedure (1) was repeated following the abrasive cleanup--the need for which was signalled by significantly high dissipation values for samples known to be of relatively low 100°C, 100 Hz dissipation. The nature and cause of the contamination buildup are not known, but presumably relate to oxidation of the cell. Nonetheless the consequences are unmistakable. One wonders how often this sort of difficulty might have led in the past to erroneous results.

Undoubtedly, other approaches could successfully be used to gain the same result here reported; the point is that it is possible, routinely, to achieve cleanliness and that reliable dissipation measurements follow as a natural consequence. At least this is the conclusion from one practical, inductive process.

ACKNOWLEDGEMENT:

Vandos Shedigian made all the dissipation measurements.

RESULTS:

Typical, replicated data obtained over a period of several days for a randomly selected sample of 1499 Pyranol (obviously of low quality) are tabulated in Table I along with calculated averages and standard deviations. Although the data are pooled, they actually come from two separate (and statistically distinct) populations: The first 13 values represent results obtained without washing the cell between measurements; the last 6 values were obtained following the previously described technique for cell cleaning. In other words, the 10% relative standard deviation is a worst-case expression of precision.

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ACKNOWLEDGEMENT:

Vandos Shedigian made all the dissipation measurements.

TABLE I

PERCENT DISSIPATION - 100°C

100 Hz	1000 Hz
2.10	0.22
2.45	.25
2.75	.29
2.15	.23
2.10	.22
2.75	.29
2.45	.25
2.70	.28
2.42	.25
2.04	.21
2.40	.25
2.60	.27
2.38	.25
2.35	.25
2.05	.22
2.21	.23
2.30	.24
2.00	.21
2.12	.22
Avg. = 2.33	0.24
Std. Dev. = 0.24	.025
% S.D. = 10.3	10.4

J.E. Fogel
7-16-68

GENERAL ELECTRIC

CAPACITOR DEPARTMENT TEST REPORT

Advance Development Laboratory SUB-SECTION NO. HF-64-062

SUBJECT

IR Spectrum of Pyranol II in 3-4 Micron Region

OBJECT

To evaluate significance of observed spectral difference between Fort Edward PII and Pilot Shop PII.

CONCLUSIONS - RESULTS - DISCUSSION

CONCLUSIONS:

The two peaks observed for PII in typical differential IR spectra in the 3 to 4 micron region are due to C-H vibrations. The longer wavelength peak (3.42 microns) is attributable to aliphatic C-H. The shorter wavelength peak (3.34 microns) has been found to correlate with titratable epoxide.

E. Howk has for some time reported a notable difference in the IR absorption spectra of Pyranol II from Fort Edward and from the Hudson Falls Pilot Shop. Two peaks are observed in the 3 to 4 micron region for the Fort Edward material, whereas the Pilot Shop PII usually shows only one peak with a small poorly resolved peak or shoulder on its low wavelength side. The longer wavelength peak (3.42 microns) has regularly been used by the Quality Control Laboratory as a measure of aliphatic(s) in PII.

The undersigned was asked to review the observations of Howk, and to attempt to elucidate the difference in the IR spectra of Fort Edward and Pilot Shop Pyranol II samples.

D. Harms, IST-G Department, M & P Laboratory, and P. Launer, Silicone Products Department, R & D Laboratory, were consulted in addition to personnel of this Laboratory. Following detailed discussions with those interested in the problem, the following hypotheses emerged as probable explanations of the observed facts:

1. The longer wavelength IR peak is due to aliphatic C-H vibration, and is a measure of added epoxide plus any other aliphatic C-H containing molecules.
2. The shorter wavelength peak is due to titratable or active epoxide C-H.
3. The appearance of the shorter wavelength peak as (1) a shoulder on the longer wavelength peak or (2) as a distinctly resolved peak depends on the relative concentrations of titratable epoxide and aliphatics, and, of course, on the effective dispersion and resolution of the IR instrument.

To test these hypotheses, the following were done:

A. Synthetic (Epoxide 206 and 1499 Pyranol) solutions were prepared by E. Howk and by R. Guiles. IR spectra were recorded, and epoxide content determined

REPORT BY: J.F. Fagel | SUBJECT CLASSIFICATION | DATE 4-23-64

TEST OR ANAL. BY:

APPROVAL:

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by H Br titration for each prepared sample.

B. The IR spectrum of cyclohexane in 1499 Pyranol was recorded in the 3 to 4 micron region.

C. Data for approximately fifty samples of Fort Edward and Pilot Shop Pyranol II were obtained from the Q.C. Laboratory files (Howk). The measured height of the shorter wavelength IR peak or shoulder was compared with the H Br titration value for percent epoxide for each sample.

RESULTS:

1. The IR spectrum of cyclohexane in 1499 Pyranol reveals a single peak (in the region of concern) at 3.42 microns. The assignment of the 3.42 micron peak to aliphatic C-H is corroborated, accordingly.

In. In the course of running the new calibration standards for percent aliphatics, it was discovered (Howk) the original calibration curve had shifted. Howk has traced the cause of the shift to increased instrumental sensitivity resulting from the addition of a new Nernst glower on March 30, 1964. (Corrections to reported percent aliphatics for the period April 1 to April 15 have been made by Howk).

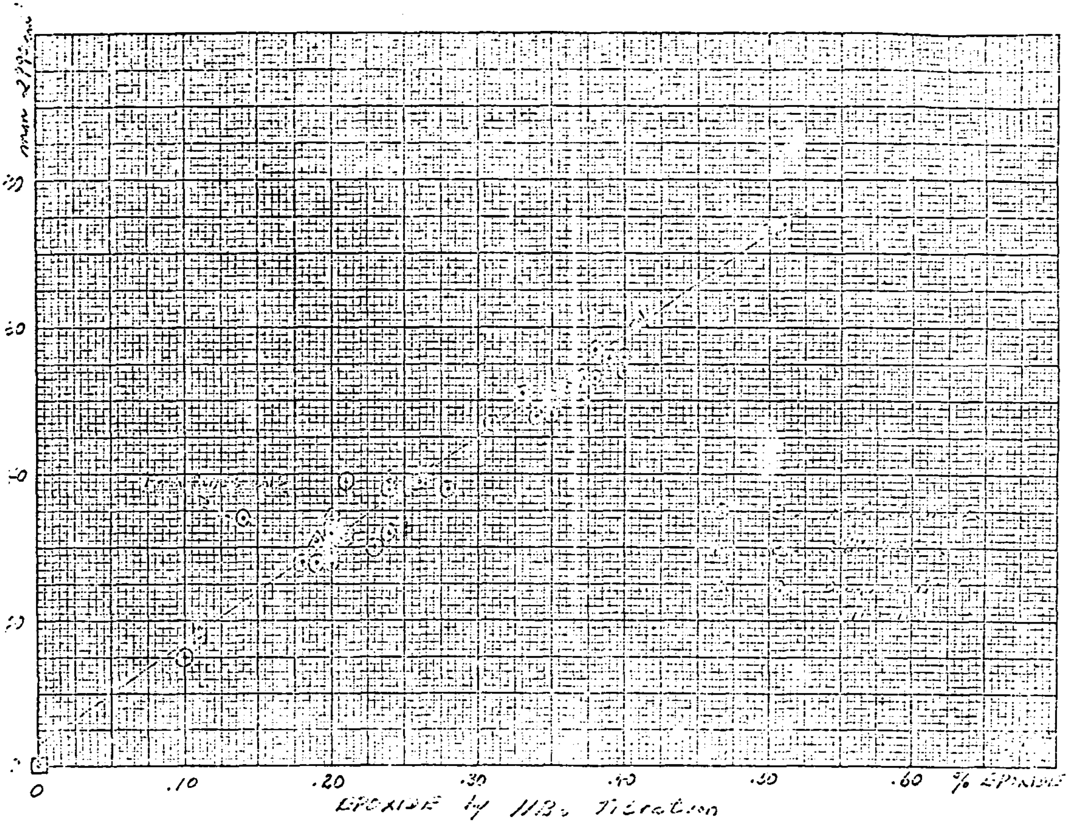
2. The measured peak heights of the shorter (3.34 micron) wavelength peak correlate linearly with titratable epoxide as illustrated in the accompanying graph. Standards made by Howk (triangles) and synthetics by Guiles (square) fit well on the straight line about which cluster the routine PII samples (circles) from Fort Edward and the Pilot Shop. (All circled values above 0.30% Epoxide represent Fort Edward PII; all below originated in the Pilot Shop).

4. Spectra recorded on the Model 521 spectrometer exhibit distinctly resolved peaks for all PII samples (lowest epoxide content 0.11%). (The Model 521 instrument gives significantly higher dispersion than the modified Model 21 that is regularly used in the Q.C. Laboratory for routine PII Analysis).

DISCUSSION:

Without attempting to elucidate fundamentals of IR absorption, it seems highly probable on empirical grounds that the cited hypotheses are correct. The correlation between peak height at 3.34 microns and percent titratable epoxide is clearly excellent for the relatively large number of samples and standards tested. Likewise, there can be little, if any, doubt of the basic correctness of the Q.C. Laboratory use of the 3.42 micron IR peak as a measure of aliphatic C-H.

GENERAL ELECTRIC COMPANY SCHENECTADY, N. Y., U.S.A.



MONS 034820

MEMO REPORT HF-64-835
November 19, 1964

EPOXIDE 206 - PYRANOL II - FULLER'S EARTH: CALORIMETRIC AND ADSORPTION STUDY

J. E. Fagel
Materials Application Unit

ABSTRACT

Calorimetric and adsorption data for Epoxide 206, Pyranol II and Fuller's Earth are reported and discussed with particular reference to the preparation of Epoxide 206 - saturated fuller's earth and the treatment of Pyranol II.

* * * *

Capacitor Department
Engineering Section
Advance Development Laboratory
Hudson Falls, N.Y.

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CONCLUSIONS

The following basic data were obtained as a result of the study aimed at learning details of the mixing of Epoxide 206 and Pyranol II with fuller's earth:

1. The specific heat (C_p) of Epoxide 206 = 0.36 cal/g°C.
2. C_p 1499 Pyranol = 0.^{P}21 cal/g-°C.
3. The heat of wetting of fuller's earth by Epoxide 206 is 280 calories/gram for slurries containing a maximum of about 14% (w/w) fuller's earth. Polymerization of the epoxide is initiated in slurries containing greater than 14% fuller's earth.
4. Thermal runaway (uncontrolled polymerization) does not occur with slurries containing as much as 28 weight percent fuller's earth. Thermal runaway cannot be initiated by preheating the Epoxide 206 before treating with fuller's earth. Thermal runaway cannot be initiated by heating a slurry to the normal boiling point of Epoxide 206 (227°C). Thermal runaway can be initiated by adding fresh Epoxide 206 to a hot slurry of reacted 206 and fuller's earth.
5. The reaction of fuller's earth with PII solutions containing 0 to 70% (w/w) Epoxide 206 follows the Langmuir adsorption isotherm.
6. Activated fuller's earth saturated with Epoxide 206 and dried by suction filtration and pressing weighs 101% more than its initial weight. Overnight vacuum drying at room temperature results in a weight loss that reduces the 101% calculated gain to 71%.
7. By HBr titration, activated fuller's earth removes up to about 15% of its own mass of Epoxide 206.
8. Fuller's earth removes Epoxide 206 from PII solutions to the maximum extent of 0.23 grams/gram.

The following conclusions are based on the results of this study:

1. The storage of thermally activated fuller's earth is troublesome: unless storage conditions are carefully controlled, the activity of the earth can be drastically changed.

2. Fuller's earth cannot by itself be used to purify Unox 206 to any gross extent (excluding the reported lowering of the acid number).

3. Fuller's earth takes up about 70% of its own mass of Unox 206 upon slurrying with the as received liquid.

4. Rapid, thorough mixing of fuller's earth and Unox 206 is essential to minimize localized reaction and consequent major heat evolution. The preferred laboratory scale order of mixing is to add liquid (one quick pour) to powder with continuous, effective stirring to achieve intimate total contact in a minimum time.

5. The slurrying of fuller's earth with undiluted Unox 206, like many chemical operations, is potentially dangerous; especially in view of our limited current understanding of the reaction(s) involved and how such reactions are initiated. Accordingly, normal safety precautions should be guaranteed by competent personnel attempting such mixing. Adequate stirring, heat dissipation, ventilation, and perhaps protective clothing are considered minimum needs.

CONCLUSIONS (cont'd.)

6. The runaway polymerization of Epoxide 206 appears to demand an acidic environment. Under no circumstance should free acid be added to Unox 206 nor should fresh Unox 206 be added to a reacted slurry of 206 and fuller's earth. (The latter probably results from improper mixing of earth and 206, and is the cause of runaway observed on some past occasions.)

INTRODUCTION

The treatment of Epoxide 206 with activated fuller's earth has resulted on occasion in thermal runaway. The work described herein was undertaken to learn some of the fundamental physical chemical facts that characterize the systems fuller's earth - Epoxide 206 and fuller's earth - Pyranol II. (Throughout this report the term Pyranol II is used to identify any concentration of Epoxide 206 in 1499 Pyranol.)

The basic information sought included measures of specific heats, heats of reaction or adsorption, the nature of the reactions (physical and/or chemical), and the relative quantities of the reactants. Reasonable answers have been obtained for the questions asked, and hopefully will be useful in further laboratory and manufacturing applications of the materials.

MATERIALS, APPARATUS, AND METHODS

To ensure reliable and representative results, care was taken to obtain and employ in this study only virgin materials typical of those used in normal Department operations.

Epoxide 206 (Vinylcyclohexene dioxide)	- An unopened 45 pound can of Unox 206 obtained from A. Abbott (can marked "3"; on Pittsfield tag 085 S/N 7912).
1499 Pyranol	- A carefully cleaned milk can was filled with refined storage liquid.
Fuller's earth	- Approximately 25 pounds of Attapulgus 60/90 mesh were obtained from a new bag supplied by C. Fuller. A small sample of 200/325 mesh material was supplied by V. Shedigian.

The activation, in small quantity lots as needed, of the fuller's earth was accomplished by heating in air at 425-450°C for four hours. Storage of the activated earth was in air at 150°C. Immediately before use, the earth was removed from the 150°C oven and cooled to room temperature in a vacuum desiccator. (Storage of the activated fuller's earth in air at 150°C is probably less desirable than storage in vacuum. Failure of the only vacuum oven available for exclusive use in this study forced the air oven storage to ensure uniformity of product throughout the prolonged experimental period.)

Active epoxide was determined in the Unox 206 and in all Pyranol II solutions by the standard HBr titration method regularly employed in the Department(4).

CONCLUSIONS (cont'd.)

Calorimetric measurements were made with a simple Dewar flask calorimeter constructed for this study. The essential components of the apparatus included a stirring assembly (both magnetic and propellar rod stirring were successfully used), thermometer, and a glass enclosed, helically wound planar heating element (fabricated to order in the Research Laboratory Glass Shop). The power input to the heating element was variable transformer controlled and measured with a GE type P-3 single phase wattmeter.

EXPERIMENTAL TECHNIQUES

The adsorption isotherm data were obtained by stirring 10 gram quantities of activated fuller's earth with 200 grams of each PII solution. Equilibration of the slurries was achieved on standing within two hours following an initial one-half hour vigorous stirring. Final titrations were performed one to two weeks following the mixing of the reactants.

The titrations were performed with about 5 gram filtered portions of supernatant liquid to determine the amount of active Epoxide removed by the fuller's earth. All samples were equilibrated and titrated at room temperature. The data were treated conventionally: the amount of Epoxide 206 removed per gram of fuller's earth was plotted against the equilibrium concentration of 206 in the PII following the reaction (Figure 1).

Calorimetric measurements were made by observing the maximum temperature reached as a result of each reaction, and then heating the cooled reaction mixture to the measured maximum temperature with the calorimeter heating element. The rate of heating was chosen to duplicate the observed rate of the studied reaction; the measured power (wattmeter) multiplied by the required time to heat the mixture to the endpoint temperature is directly equal to the energy of the reaction.

Initial calibration of the Dewar flask calorimeter (necessary to enable calculation of the specific heats of Epoxide 206 and 1499 Pyranol) was accomplished by measuring temperature increases upon dilution of aqueous sulfuric acid solutions. The difference between observed and calculated temperature increases is a measure of heat loss to the calorimeter.

RESULTS

1. Specific heat determinations at atmospheric pressure.

   A. Epoxide 206

      199 grams, 25.5°C to 84.4°C, 6.4 kcal.; loss to calorimeter - 2.2 kcal

      $$C_p = \frac{(6.4-2.2)}{(199)(84.4-25.5)} = 0.36 \text{ cal./g-deg C.}$$

   B. 1499 Pyranol

      199 grams, 25.3°C to 89.1°C, 4.9 kcal.

      $$C_p = \frac{(4.9-2.2)}{(199)(89.1-25.3)} = 0.21 \text{ cal./g-deg C.}$$

RESULTS (cont'd.)

2. Percent active or titratable epoxide in Unox 206.

The average of several determinations made over a period of many weeks is 95.3% by weight. No attempt was made to isolate or to identify the non-titratable constituent or constituents.

3. Adsorption Isotherm.

The basic data characterizing the reaction of activated fuller's earth and PII solutions are tabulated in Table I and shown graphically in Figure 1. Each datum point was obtained with an equilibrated slurry of roughly 10 grams of fuller's earth and 200 grams of PII solution.

The straight line plot indicates the data conform to the Langmuir equation that describes the adsorption of a monolayer on a crystalline solid. The calculated constants for the Langmuir equation, obtained by measuring the slope and intercept of the straight line, are as follows:

$$a = 7.0 \times 10^{-4}$$

$$b = 240$$

The Langmuir equation is expressed in linear form as:

$$\frac{C}{(x/m)} = \frac{1}{b} C + \frac{1}{a}$$

c = the equilibrium concentration of the adsorbed species
x = mass of adsorbed species taken up by the adsorbent
m = mass of the adsorbent
a,b = constants characteristic of a particular adsorption system (b is proportional to the number of gram moles of adsorbed species for square centimeter of adsorbent).

4. Saturation of fuller's earth with Epoxide 206.

A. Mass Increase of fuller's earth.

38.1 grams of activated fuller's earth were slurried with 200 ml. of Epoxide 206 in an ice bath. The solution was stirred continuously for about 20 minutes and then allowed to stand undisturbed about one hour. The solids were then collected by suction filtration, and pressed between sheets of dry filter paper to remove excess liquids. The product finally obtained was apparently dry, and had a mass of 75.3 grams. After standing overnight at room temperature in an evacuated vacuum desiccator, the mass of the saturated earth was approximately 65 grams. The mass increase of the fuller's earth following saturation by Epoxide 206 equals:

$$\frac{75.3 - 38.1}{38.1} = 97.6\%$$

or, after vacuum drying,

$$\frac{65 - 38}{38} = 71\%$$

RESULTS (cont'd.)

Subsequent vacuum drying (an additional 24 hours) did not further reduce the mass of the saturated earth. Repeated determinations of the mass increase of fuller's earth upon saturation with Epoxide 206 gave results of 101.6% and 102.6%; thus yielding an average mass increase of 100.6%.

B. Loss of Titratable Epoxide Following Saturation of fuller's earth.

(a) 198 grams of 206 were slurried at room temperature with 50.2 grams of fuller's earth. After equilibration, the filtered supernatant liquid was found by HBr titration 92.1% active epoxide. The active epoxide lost was accordingly: $(95.3\%-92.1\%)(198)=6.3$ grams or $\frac{(6.3)}{50.2}$ 100 = 12.6% of mass of fuller's earth.

(b) 201 grams of 206 were similarly slurried with 61.0 grams of fuller's earth. The epoxide lost was:

$(95.3\%-90.6\%)(201) = 9.5$ grams

or $\frac{(9.5)}{61.0}$ 100 = 15.5% of mass of fuller's earth.

(c) 199.9 grams of 206 were similarly slurried with 41.0 grams of fuller's earth. The epoxide lost was:

$(95.3\%-94.8\%)(199.9) = 1.0$ grams

or $\frac{(1.0)}{41.0}$ 100 = 2.4% of mass of fuller's earth.

5. 1499 Pyranol - Epoxide 206 Saturated fuller's earth.

The possible desorption of active 206 from fuller's earth by 1499 Pyranol was examined by slurrying 10 grams of the saturated earth with 100 ml of 1499. After standing overnight, portions of the liquid were titrated by the standard HBr method. Results are tabulated below:

Blank Titration 1499 Pyranol - 0.00% Epoxide

Fuller's earth	1499	% Epoxide in Equilibrated 1499
10 g	100 ml	0.75
10 g	100 ml + 500 ppm H_2O	0.72
10 g*	100 ml	0.42
10 g*	100 ml + 500 ppm H_2O	0.44

*Stored about 48 hours in vacuum desiccator.

6. Calorimetric Measurements.

The bulk of the data obtained with slurries of Epoxide 206 and fuller's earth are summarized in Table II and plotted in Figure 2.

RESULTS (cont'd.)

Data obtained for PII solutions mixed with fuller's earth fit in well with the results for the undiluted 206 and fuller's earth as noted.

The effect of particle size of the fuller's earth was observed in one experiment in which a sample of 200/325 mesh earth was used instead of the usual 60/90 mesh material. (Insufficient material was obtainable for any further work.) The heat evolved was significantly greater than for comparable experiments with 60/90 mesh fuller's earth, but not proportional to the calculated surfaces ratio:

60/90 mesh - average particle diameter about 200 microns
200/325 mesh - average particle diameter about 50 microns

assuming spherical particles and constant density, the ratio of surface areas for equal masses of the two materials is $\frac{(200)^3}{(50)^3} \cdot \frac{(50)^2}{(200)^2} = 4$

The ratio of heats evolved was 3.7/2.6 = 1.4.

Several attempts were made to initiate the runaway polymerization of the epoxide; all failed except two. Preheating the Epoxide 206 prior to mixing with fuller's earth resulted in no greater heat evolution than observed for comparable quantities of room temperature mixed reactants. Preheating was tried from 50°C to the normal boiling of the epoxide (227°C) without effect.

Heating of the reaction slurry to temperatures as high as 227°C likewise had no measurable effect on the apparent nature or extent of the reaction.

The order of mixing the reactants also had no apparent effect (contrary to Shedigian's experience - cf. 2). Comparable heat evolution was measured for slurries made by adding liquid to solid and by adding solid to liquid.

Thermal runaway, resulting in the violent boil off of liquid and the production of a nearly intractable mass, occurred only twice in the course of this work. Both instances (one unplanned, the other deliberate) had a common background: fresh Epoxide 206 was added to a hot slurry of reacted 206 and fuller's earth.

In the planned experiment, 10 grams of activated fuller's earth were slurried with 200 grams of Epoxide 206 preheated to 125°C. After one hour, the temperature of the slurry had dropped (in steady fashion) to 87°C. At this stage it appeared nothing was likely to happen, so heat was applied to raise the temperature to 150°C, then to 200°C, and subsequently to the boiling point with a holding period at each temperature of several minutes. At no time during the experiment was evidence obtained that indicated any reaction beyond the initial adsorption illustrated in the tabulated data; that is, as soon as heat was no longer applied from the calorimeter heater, the temperature of the slurry started to decline.

RESULTS (cont'd.)

After approximately 20 minutes, following unsuccessful thermal attempts to initiate polymerization of the epoxide, 25 grams of fresh room temperature Epoxide 206 were added to the slurry at a temperature of 220°C. Immediately the slurry temperature dropped to 200°C, and then gradually rose over the next hour to a maximum of 273°C. On subsequent inspection of the contents of the calorimeter, only a solid, dark brown mass was observed. The polymerized epoxide fused the fuller's earth particles into the nearly intractable material mentioned earlier. (Equipment cleanup was achieved only by soaking in hot caustic and by mechanically chipping away the "cement.") The reaction terminated short of complete violence only because the excess liquid reactant boiled off before it could react.

Continued boiling of Epoxide 206 in air also fails to result in uncontrolled polymerization. Treatment of 206 with aqueous potassium hydroxide leads to the formation of yellowish, gelatinous material after many hours, but nothing more.

The addition of aqueous sulfuric acid (concentrated) to excess Epoxide 206 yields within a few minutes red colored, gelatinous material. Shortly after the formation of the gel, a violent exothermic reaction suddenly occurs leaving only a black (largely carbon) residue. The addition of dilute aqueous sulfuric acid to excess Epoxide results in an instantaneous explosive reaction and the formation of a resinous material.

DISCUSSION

The data of Table I are at least prima facie evidence that the reaction between fuller's earth and Pyranol II solutions is strictly physical - as one might predict. Saturation occurs at equilibrium concentrations greater than about 14% Epoxide 206 and corresponds to 0.23 grams of epoxide per gram of fuller's earth. From the shape of the curve, it is evident only the Epoxide 206 is adsorbed from the solutions, and there is no desorption of active epoxide by 1499 Pyranol over the concentration range 0 to 70% Epoxide 206.

The saturation of fuller's earth by Epoxide 206 is difficult to evaluate uniquely as evidenced by the variation in results obtained by different techniques: 100% to 70% approximate takeup by mass increase measurement, 2.4% to 15.5% by titration, and 23% from the adsorption isotherm. It seems reasonable to assume the unusually high value obtained by mass increase determination might be a consequence of the presence of excess Epoxide 206 in the saturated earth despite best attempts to dry. It is also possible that the temperature control (ice bath) used during the mixing of the 206 and fuller's earth for the mass gain determination may have influenced the adsorption sufficiently to account for the observed result.

From the standpoint of reconciliation of results, the 100% and 70% values might be dismissed; they have practical utility, however, from the point of view of the preparation of saturated earth for manufacturing application. (See added discussion in addendum.)

DISCUSSION (cont'd.)

The difference between the isotherm value (23%) and the direct titration values for raw Epoxide 206 and fuller's earth is not readily explained. Aside from the evidence presented by the isotherm (that 1499 is not adsorbed from PII solutions), one would predict the effect, if any, of 1499 would be to interfere with and lessen the adsorption of Epoxide 206 by fuller's earth. Actually, the opposite appears to be the case; but the results may be confounded by the temperature difference during slurrying as noted earlier. (See addendum.)

The results of the experiment aimed at elucidating the response of Epoxide 206 saturated fuller's earth to 1499 Pyranol with and without added water are also partly indeterminant. From the only data currently available, one cannot tell whether 1499, with or without added water, strips little epoxide from the saturated earth or whether possibly stripped material is no longer HBr titratable. It is possible the adsorption of Epoxide 206 by fuller's earth is non-reversible, but it may be the present data should be interpreted simply as indicating Epoxide 206 is tenaciously held by fuller's earth and cannot be stripped by 1499 Pyranol and a small amount of water. These same data also shed additional light on the interpretation of the takeup of Epoxide 206 during saturation of fuller's earth.

Assuming the previously discussed 70% takeup figure is erroneously high because of occluded free Epoxide 206, and the correct measure of adsorbed epoxide is approximately 20% of the mass of fuller's earth saturated, one calculates that 10 grams of vacuum dried saturated earth contains about 2 grams of free Epoxide 206. Such free Epoxide 206 should be readily soluble in 1499 Pyranol, and the concentration of epoxide in the equilibrated 1499 should be at least 1.4% instead of the measured 0.4%. The assumption of a significant amount of mechanically held Epoxide 206 in saturated fuller's earth is accordingly untenable, and one should perhaps not be hasty in dismissing the 70% takeup value.

The calorimetric data are essentially straightforward and agree well with the usually noted values for heats of wetting or adsorption up to the 14% fuller's earth concentration. The differences in observed heat evolutions and the extrapolated heat of adsorption above the 14% point are of the correct order for heats of polymerization (roughly 10 kcal/mole). That the large amount of heat released, especially in runaway situations, is attributable to polymerization of the epoxide is only conjectural. The evidences, coupled with possible reactions, however, suggest there is little likelihood this conjecture is incorrect. The real question is what causes the runaway condition that is occasionally encountered?

The basic facts seem to dictate acidic catalization as necessary for polymerization of Epoxide 206. To weave this in with the observation that runaway occurs when fresh Epoxide is added to a hot slurry of reacted fuller's earth and 206 is not immediately straightforward. Among other possibilities, suspicion is directed toward the roughly 5% of non-titratable constituent(s) in as received Unox 206, toward conceivable reaction product(s)

DISCUSSION (cont'd.)

resulting from admixture of 206 and fuller's earth, and toward some interaction between these constituents and products. Reacted slurries are indeed mildly acidic as determined by aqueous extraction and conventional pH determinations. If acidity is the key, why then is it necessary to add fresh Epoxide 206 to a slurry already containing a gross excess of 206 before runaway polymerization occurs? A possible explanation lies in the reported lowering of the acid number of Unox 206 following fuller's earth treatment. The reacted slurry may have lost acidic contaminants present in the original Unox 206 to such an extent that polymerization is not possible; the addition of fresh 206 could, however, provide sufficient acid to establish the final necessary condition for runaway polymerization.

Obviously, little is contributed by continued conjecture concerning a limited store of facts. Further work is clearly dictated to elucidate the details of the runaway reaction. In the interim it appears safe to conclude that any practical application of the subject materials should be cautiously steered around the condition of fresh Epoxide 206 being added to a hot, reacted slurry of 206 and fuller's earth. Without regard to the mechanical details involved (which of course can be limiting), the preferred method of slurrying fuller's earth and Epoxide 206 seems to be to add the liquid quickly to the solid rather than the solid to the liquid (with apologies again to Shedigian, 2). Rapid mixing of the bulk of the two materials is the essence of the technique required to avoid localized effects that often lead to partial fuming and charring if not full blown runaway. The transfer of the liquid rather than the powder tends to minimize localized effects better than the opposite approach - at least on the laboratory scale here reported. External cooling to limit the temperature rise of the slurry is of course immediately suggested as a necessary adjunct to the safe handling of quantity lots of the materials. The calorimetric data should permit reliable estimation of the required cooling capacity for a desired operation. It is a moot point, however, whether or not external cooling can fully arrest polymerization once initiated.

Finally, some comment on the effective activity of any batch of fuller's earth is in order. The bulk of the work reported here was done during the period of July through August, and many small batches of fuller's earth were activated and stored as earlier described. On several occasions results were obtained that clearly indicated the activity of the earth is particularly sensitive to time and conditions of storage following initial high temperature activation. Storage in a good vacuum oven at perhaps 100°C would be the writer's choice to ensure optimum activity of the earth. Special care would be required, however, in handling such material to avoid certain, probably rapid deterioration on exposure to air (particularly during high humidity periods). As noted, air storage at 150°C was employed for the activated fuller's earth used in this study; it is considered that all the data here reported may likely have been significantly (but uniformly) influenced by this storage. That is, greater adsorption and increased heats of wetting may well be observed for comparable reactions involving activated fuller's earth stored in vacuum above room temperature, and exposed to air for a minimum time prior to reaction.

CONCLUSIONS

Much remains that can be done to further elucidate the nature of the studied reactions. The question is whether or not the information gained would be worth the required efforts. For the moment, at least, the writer is of the opinion that enough time has already been spent and any further effort should be dictated by a specific, practical need involving a particular feature of the basic reactions.

Further titration data and a one shot infrared absorption analysis hopefully can be added in addendum to aid in answering the question posed regarding the reaction between Epoxide 206 - saturated fuller's earth and 1499 Pyranol. Aside from this eleventh hour burst, the study is probably best considered closed.

ADDENDUM

The following data were obtained after the writing of the main text was completed.

Saturation of Fuller's Earth by Epoxide 206

50.0 grams of fuller's earth were slurried with 200.5 grams of Epoxide 206 in an ice bath. After standing overnight, the percent active epoxide in the supernatant liquid (as determined by HBr titration) was only 0.4% less than in the original Unox 206 - corresponding to less than 2% takeup by the fuller's earth. (It is highly doubtful the observed 0.4% difference is significant. Accordingly there is no real evidence of any preferential takeup of active epoxide in this experiment - or in the earlier noted experiment in which a calculated 2.4% takeup was recorded.)

In another preparation of Epoxide 206 - saturated fuller's earth, an excess of 206 was slurried with an unknown mass of fuller's earth. After overnight standing, the percent active epoxide in the supernatant liquid was found by titration 5% less than in the starting Epoxide 206.

The four separate titration determinations of loss of active epoxide following saturation of fuller's earth will be noted to constitute two pairs: (1) about 1½% loss and (2) about 0% to 2% loss. It will further be observed that the 0% to 2% loss pair were slurries that experienced relatively small temperature rises (one because of ice bath cooling, the other because the proportions of fuller's earth and Epoxide 206 yielded little more than the heat of wetting), whereas the 1½% average loss pair originated from slurries that reached temperatures at least 100°C (because the percentage fuller's earth in each slurry was well above the level at which polymerization of the epoxide is initiated - Figure 2).

One can conclude from these observations that fuller's earth does not selectively adsorb active epoxide from Unox 206, and that the titration loss measured for slurries that have been relatively hot is due not to preferential adsorption of active epoxide but to loss of active epoxide through partial polymerization.

The final experiment involved the mixing of 10 grams of Epoxide 206 - saturated fuller's earth with 100 grams of 1499 Pyranol. After overnight standing, aliquots of liquid were taken for HBr titration and for infrared absorption analysis. The results are tabulated below:

% Epoxide by titration:	1.26%; 1.27% (w/w)
% Epoxide by IR:	1.29%; 1.30%
% Aliphatics by IR:	1.33%; 1.34%

The difference between active epoxide and percent aliphatics is most likely not significant, and accordingly suggests there is no stripping of inactive epoxide from the fuller's earth by 1499 Pyranol. The relatively high values (1.3%) undoubtedly were a result of the use of saturated earth that had been suction filtered and pressed but not vacuum dried; that is, excess free

APPENDIX (cont'd.)

epoxide was obviously present in the earth when slurried with the Pyranol. If one in fact assumes the 1.3 grams of titratable epoxide represent mechanically held 206, the calculated amount of Epoxide 206 lost on slurrying with fuller's earth is 7% of the mass of the earth (mass increase of earth after slurrying = 100%; therefore 10 grams of saturated earth contains 5 grams of earth plus 5 grams of contained Epoxide 206 - but 1.3 grams of the 5 grams of 206 are soluble, and titratable in 1499 Pyranol; 3.7 grams of 206 are therefore held by the fuller's earth). The agreement between this 7% and the mass increase value for vacuum dried saturated earth is at least an interesting coincidence!

The final conclusions based on all the reported results is that fuller's earth non-selectively sorbs and tenaciously retains about 70% its own weight of Unox 206. The non-selective sorption leads to no change in composition of the 206, and accordingly the apparent discrepancy between mass increase and titration determinations of 206 uptake by fuller's earth is explained. The titration change noted in some cases is the result of loss of active epoxide through polymerization.

The sorption of Epoxide 206 from 1499 Pyranol solutions is selective as evidenced by the adsorption isotherm data (Table I - Figure 1). Why the sorption is selective from solutions and non-selective from undiluted Unox 206 is not obvious. It seems obvious, however, that one cannot expect to purify Unox 206 simply by equilibration with fuller's earth.

ACKNOWLEDGMENT

All of the experimental work here reported was performed by A.J. Munichiello whose assistance is gratefully acknowledged.

A. Katchman provided details of related unpublished work, and other helpful information and advice.

R. Guilen performed the IR analyses.

REFERENCES

1. Test Report HF-63-67, P. Scherer, Stock Qualification Data for Epoxide 206.
2. Test Report HF-64-1007, V. Shedigian, Purification of Epoxide 206 with Activated Fuller's Earth.
3. Memo Report HF-63-931, A. Katchman, Epoxide Stabilization of Capacitors.
4. Durbetaki, A.J., Anal. Chem., 28, 2000 (1956).

TABLE I

ADSORPTION ISOTHERM

C(g/100 g)	x/m (g/g)	$\frac{C}{x/m}$ (g/g)
0.0	0.0	-
0.4	0.17	2.4
0.5	0.10	5.0
0.5	0.09	5.6
2.3	0.14	16.4
4.0	0.18	22.2
4.3	0.19	22.6
5.7	0.22	25.9
6.5	0.19	34.2
8.8	0.21	41.8
9.2	0.17	54.0
10.4	0.25	41.6
13.1	0.23	57.0
13.3	0.23	57.9
13.3	0.25	53.2
13.6	0.23	59.2
15.1	0.22	68.7
22.6	(0.32)?	(70.6)?
22.7	0.24	94.8
46.0	(0.43)?	(107.)?
46.4	0.23	202.
69.8	0.21	332.

TABLE II

CALORIMETRIC MEASUREMENTS
(Epoxide 206 + fuller's earth)

% Fuller's earth	kcal Evolved	Time to Reach Temperature Max.
4.60	1.32	1800 secs.
4.86	1.24	900
5.15	1.76	9900
6.90*	1.44	420
7.03*	1.50	13100
10.3	3.16	2640
12.8*	3.60	2580
13.5	5.38	>2800
13.8	4.05	1100
17.0	5.67	660
20.2	9.14	540
22.0	11.72	420
23.3	10.85	400
24.6	10.85	360
27.6	15.1 ✓	-
9.2^	3.74	

*PII solutions, about 71% by weight Epoxide 206 (% f.e. relative to Epoxide 206, not to PII).

✓ Calculated from C_p data and observed temperature maximum of reaction.

^200/325 mesh fuller's earth.

MR HE-64-835

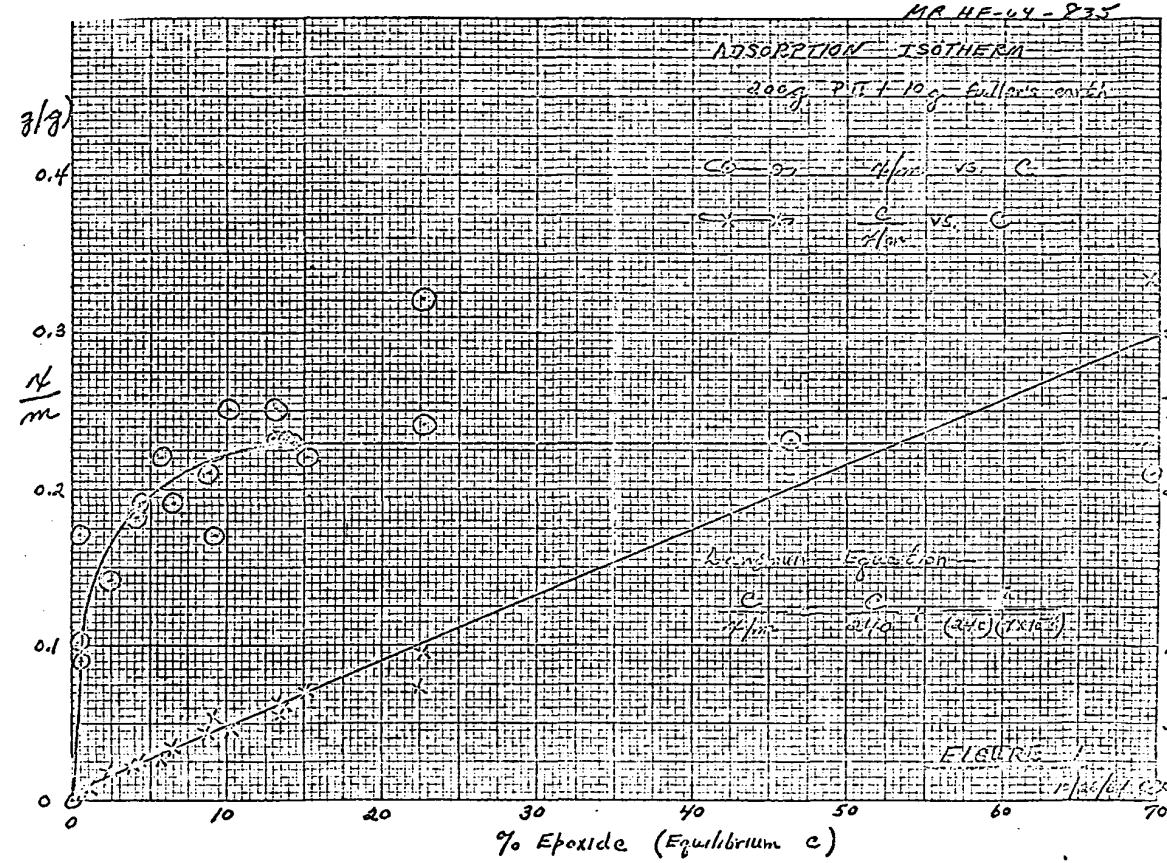

FIGURE 1

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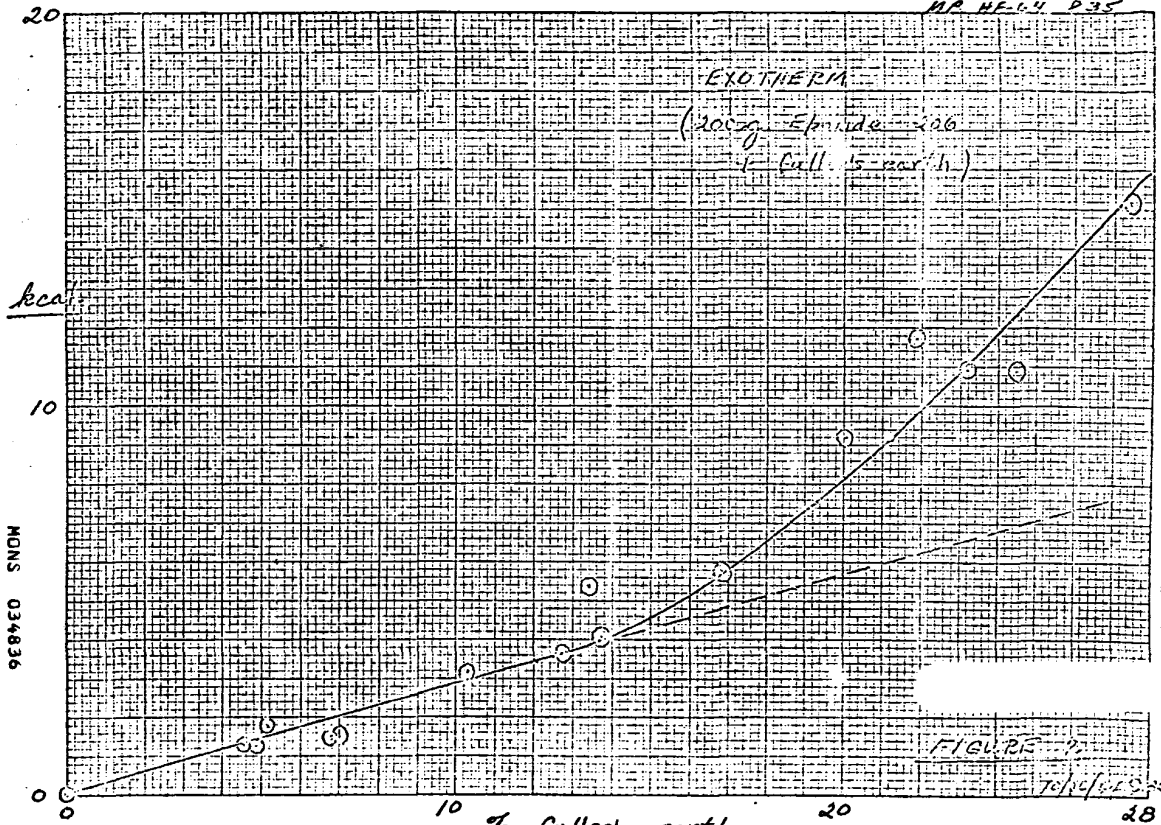

FIGURE 2

MATERIAL SPEC.

GENERAL ELECTRIC

A1381-S7

SPECIFICATION

CHLORINATED DIPHENYL LIQUID INSULATION

Supersedes A13B1-S6

G-E Material A13B1 identifies chlorinated diphenyl liquid insulation, as follows:

A13B1 - 12 C pour point
A13B1C - Minus 14 C pour point
A13B1E - 25 C pour point

PROPERTIES:

	A13B1	A13B1C	A13B1E
Specific gravity at 65/15.5 C	1.495-1.505	1.381-1.392 (1)	1.555-1.566 (2)
Refractive index at 25 C	1.6370-1.6390	1.6240-1.6260	1.6455-1.6475
Total acid No., mg KOH/g, max	.010	.010	.014
Free chlorides, ppm, max	NDA (3)	.05	NDA (3)
Sulfates	None	None	-
Water, ppm, max	35	35	35
Viscosity, Saybolt Universal, seconds:			
At 37.8 C	-	82-92	-
At 98.9 C	44-48	-	72-78
Distillation range, deg C:			
10% distilled	366-378	325 min	380-388 (4)
50% distilled	371-383	-	390-404 (4)
90% distilled	379-394	350 max	400-420 (4)
Pour point, deg C, max	12	-14	25-34
Flashpoint, deg C	-	170-200	-
Fire point, deg C, min	None	None	350
Color, max	40	40	150
Dielectric strength, 25 C, KV, min	35	35	35 (5)
Dielectric constant at 100 C	4.15-4.35	4.70-4.90	-
Resistivity at 100 C, ohm-cm x 10^9, min	500	500	500
Dechlorination, ppm, max	5.0	1.0	-
Corrosion stability - After a sample containing one gram of aluminum foil is heated for six hours at 210 C, the aluminum foil shall be free from corrosion as determined visually or by loss in weight and the sample shall conform to the following requirements:			
Total acid No., mg KOH/g, max	-	-	.014
Free chlorides, ppm, max	-	-	NDA (3)
Color, max	-	-	150
Condition	-	-	Clear

(1) At 25/15.5 C.
(2) At 80/15.5 C.
(3) No de ectable amount.
(4) % distilled determined on a weight basis.
(5) At 60 C.

MANUFACTURE:

Material - Shall be a chlorinated diphenyl liquid.
Impurities - Material shall be clean and free from all injurious impurities such as metallic or non-metallic particles or other contamination.
Approval - After a material has been approved under this specification, no change in manufacturing or refining methods shall be made without prior approval of the purchaser.

TESTING METHODS:

Sampling	ASTM D 923-56
Specific gravity	ASTM D 1481
Refractive index	ASTM D 1807-58
Acid number	ASTM D 974-58 (6)
Free chlorides (A13B1 and A13B1E)	G-E Procedure
(A13B1C)	G-E Procedure
Sulfates	ASTM D 1275-61
Water	G-E Procedure
Viscosity	ASTM D 445

(6) Except use 0.01 N aqueous KOH.

(Continued on page 2)

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MATERIAL SPECIFICATIONS (Continued)

Distillation	ASTM D20-56 (7)
Pour point	ASTM D97-57 (8)
Flash & fire point	ASTM D92-57
Color	ASTM D801-56
Corrosion stability	G-E S4C26
Dielectric strength	ASTM D877-49
Dielectric constant	ASTM D924-49
Resistivity	G-E S4D2
Dechlorination	G-E S4C48

(7) Except that percent distilled is determined on a weight basis with the following modifications in procedure:

Correct results for emergent thermometer stem and barometric pressure. For A13B1E only use a heated condenser consisting of a center glass tube 5/8 inch ID by 3/4 inch OD by 26 1/4 inches long, cut off at 45° slant on lower end. Wrap for 18 inches with 48 turns spaced 3/8 inch on center with flat Nichrome wire .005 x 1/8 inch. Bring out ends of wire through holes in outer jacket. Outer glass jacket is 1 3/8 inches OD and is sealed to center tube at upper end. Lower end is drawn down to fit snugly against center tube but is not sealed to it. The condenser heat is controlled by a Variac to an internal temperature of 125 ± 10 C which is started when the heat to the flask is turned on. Adjust the distillation rate to 50-70 drops per minute and collect the distillate in a tared flask set on a beam balance.

(8) Except using only a single bath continually cooled to pour point.

SAMPLE APPROVAL: (A13B1 and A13B1C only)

The supplier shall forward to the laboratory of the purchaser at the point of delivery a two quart sample taken from each manufacturing batch of this material scheduled for shipment to the General Electric Company or others designated by the General Electric Company. This sample shall be designated as "Advance Sample, Lot No.____, G-E Material_____".

Approval of this advance sample by the General Electric Company does not constitute approval of the shipment. General Electric Company approval will be based upon the material as received at its destination and will be based not only on conformity to the properties specified herein but on conformity to all performance characteristics deemed essential by the purchaser.

CERTIFICATE OF TEST:

The vendor shall submit promptly to the laboratory of the purchaser at the point of delivery a certificate of test in duplicate so that it will be received on or before receipt of shipment. The certificate of test shall include the following information.

Specific gravity	Water content
Color	Dielectric strength (A13B1 & A13B1C only)
Condition	Dielectric constant (A13B1 & A13B1C only)
Acid number	Resistivity (A13B1 & A13B1C only)
Distillation range	Pour point
Refractive index	Viscosity (A13B1E only)
Free chlorides	Flash point (A13B1C only)
Corrosion stability (A13B1E only)	Dechlorination (A13B1 & A13B1C only)

This certificate shall also contain the G-E designation, purchase order number, tank car number, lot number, and date shipped, so that the certificate may be identified with the shipment.

PACKING AND MARKING:

Material shall be shipped in tank cars or drums, as specified on the purchase order.

All containers shall conform to ICC regulations and shall be of a type having no deleterious effect on the material. Extreme care shall be exercised by the supplier in the selection and use of vacuum cleaner containers or containers provided by the supplier. Such containers shall be carefully inspected by the supplier to assure that they are free of injurious foreign matter.

Adequate precautions shall be taken by the supplier to assure retention of the properties and characteristics of the material, as specified herein, upon arrival of the shipment at point of delivery.

Each container shall be legibly marked with the purchase order number, the manufacturer's name, the G-E designation and the lot number.

GENERAL ELECTRIC

CAPACITOR DEPARTMENT
SPECIFICATION
FOR
PYRANOL II

A50PC8-S3

Orders and Correspondence Must Specify Complete Number

February 11, 1965
Supersedes A50PC8-S2

G-E Material identifies epoxide modified chlorinated diphenyl liquid insulation as follows:

A50PC8A - Pyranol II - Epoxide 0.35%
A50PC8B - Pyranol II - Epoxide 0.25%
A50PC8C - Pyranol II - Epoxide 1.00%

PROPERTIES:

	A50PC8A	A50PC8B	A50PC8C
Specific Gravity	1.381 - 1.392	1.381 - 1.392	1.381 - 1.392
Refraction Index	1.6240 - 1.6260	1.6240 - 1.6260	1.6240 - 1.6260
Total Acid No.,mg KOH/g, (max)	.05	.05	.05
Water Content, PPM, Max	20	20	20
Sulfates	None	None	None
Thermal Stability Chlorides (2 hrs. at 240 C) PPM, max	NDA	NDA	NDA
Active Epoxide Content - % by weight	0.35±.05	0.25±.05	1.00±.10
Dielectric strength, P5C, KV, min	35	35	35
Dielectric Constant at 100 C	4.70 - 4.90	4.70 - 4.90	4.70 - 4.90
Resistivity at 100 C, ohm-cm X 10^9, min	1100	3500	1100
Pour Point deg. C, max	-14	-14	-14
Flashpoint deg. C	170 - 200	170 - 200	170 - 200
Fire Point	None	None	None
Aliphatic Content, max % -Actual Active Epoxide Content % plus	.07%	.05%	.15%

MANUFACTURE:

Material - Shall be a blend of chlorinated diphenyl (A13B1C) and epoxide (D5D71A)

Impurities - Material shall be clear and free from all injurious impurities such as metallic or non-metallic particles or other contamination.

REFERENCE METHODS:

Sampling	ASTM D901-56
Specific Gravity	ASTM D901-56
Refractive index	ASTM D901-56
Acid number	ASTM D974-55 (1)
Sulfates	ASTM D878-49
Free Chlorides	G-E EAch1C
Water	G-E EAch45
Pour point	ASTM D97-57 (2)
Flash and fire point	ASTM D92-57
Dielectric strength	ASTM D877-49
Dielectric constant	ASTM D924-49
Resistivity	G-E EAD2
Active Epoxide content	G-E EAc50
Aliphatic content	G-E E50PC25

(1) Except use 0.01 N aqueous KOH
(2) Except using only a single bath continually cooled to pour point

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MDSink

GENERAL ELECTRIC

SPECIFICATION

VINYLCYCLOHEXENE DIOXIDE

DO NOT SEND OUTSIDE GENERAL ELECTRIC COMPANY

G-E Material D5D71 identifies vinylcyclohexene dioxide, as follows:

D5D71A - General purpose

PROPERTIES:

Specific gravity, 20/20 C	1.08-1.10
Viscosity at 25 C, centistoke, max	[illegible]
Total epoxide (as oxirane oxygen), %, by weight, min	21.7
Acid number, %, max	.05
Iodine number, max	5.0
Color (platinum - cobalt), max	100
Suspended matter	Substantially free

MANUFACTURE:

Material - Shall be a vinylcyclohexene dioxide.

Impurities - Material shall be clear and free from all injurious impurities as metallic or non-metallic particles or other contamination.

Approval - After a material has been approved under this specification, no change in manufacturing or refining methods shall be made without prior approval of the purchaser.

REFEREE METHODS:

Sampling	ASTM D[illegible]
Specific gravity	ASTM D1310
Viscosity	ASTM D445
Color	ASTM D[illegible]

Acid number:

Introduce 60 gm of the sample weighed to the nearest 0.1 gm into a 250-ml Erlenmeyer flask containing 50 ml of methanol which has been previously neutralized to a faint pink end point using 1.0 percent phenolphthalein indicator in methanol. Swirl to effect solution.

Add a few additional drops of the indicator and titrate with standard 0.1 N alcoholic potassium hydroxide to the first pink end point permanent for at least 15 seconds.

Calculation:

$$\frac{AN \times 6.0}{gm\ sample} = \text{acidity, \% by weight as acetic acid}$$

A = ml of N Normal KOH required

Total epoxide:

CAUTION: This procedure involves the use of perchloric acid. DO NOT use this reagent unless aware of its potential hazards.

Hydrogen bromide solution, 0.5 N in acetic acid:

By means of a graduate carefully add 67 ml of cp bromine to two liters of glacial acetic acid. Add reagent grade phenol in 10-gm increments until the solution becomes light straw in color. (approximately 100 gm) and add 10 gm in excess. Mix the soltuion after each addition of the phenol and allow the final solution to stand overnight before using.

0.2 N sodium acetate in acetic acid:

Dissolve 16 gm of anhydrous sodium acetate in sufficient glacial acetic acid to make one liter of solution. Standardize this solution against standard 0.2 N perchloric acid using 1.0 percent crystal violet indicator in acetic acid.

Procedure:

Prepare a sufficient number of clean dry 250-ml iodine flasks to perform all sample and blank determinations in duplicate.

Carefully pipet 25 ml of the hydrogen bromide solution into each of the flasks, using the same pipet for each transfer.

Reserve two of the flasks for the blank determination.

Into each of the other flasks introduce 0.3 to 0.4 gm of the sample weighed to the nearest 0.1 mg by means of a suitable weighing pipet and swirl to effect solution.

Stopper the flasks using 5 ml of glacial acetic acid as the liquid seal and allow the samples to stand with the blanks at room temperature for 15 minutes.

 (Continued on page 2)

REFEREE METHODS: (Continued)
Total epoxide: (Continued)
Procedure: (Continued)
Carefully remove the stopper from each flask and wash down the stopper and inside walls of the flasks with 25 ml of glacial acetic acid.
To each flask add 5 or 6 drops of crystal violet indicator and titrate with standard 0.2 N sodium acetate in acetic acid to the first bluish-green end point.
Calculation:

$$\frac{(B - A)N \times 1.6}{\text{gm sample}} = \text{total epoxide, \% by weight, as oxirane oxygen}$$

A = ml of N normal sodium acetate required for the sample
B = average ml of N normal sodium acetate required for the blank

Iodine number:
Wijs solution:
Dissolve 13 gm of resublimed iodine in 1000 ml of glacial acetic acid. Gentle heat may be necessary to effect solution. Allow to cool and remove approximately 200 ml of the solution. Pass dry chlorine gas into the remainder of the iodine solution until the original titration is not quite doubled. Perform the titration as directed below. A characteristic color change takes place in the Wijs solution when the desired amount of chlorine has been added. This may be used as an aid in judging the end point. Add a small excess of chlorine and bring back to the desired titration by addition of some of the original iodine solution.
Into respective 500-ml Erlenmeyer flasks pipet 25 ml of the solution before addition of the chlorine and 25 ml of the solution after addition of chlorine. Add 20 ml of 15 percent potassium iodide solution in distilled water and 100 ml of distilled water to each flask. Titrate the contents of each flask with standard 0.1 N thiosulfate until the yellow color almost disappears. Add two ml of 1.0 percent starch indicator solution and continued titrating until the blue color disappears.
Procedure:
Heat all samples which are solid or semi-solid or contain turbidity under an infrared heat lamp or in a hot water bath until completely homogeneous, and mix well.
Prepare a sufficient number of clean dry 500-ml glass-stoppered Erlenmeyer flasks to perform all blank and sample determinations in duplicate.
To each flask add 20 ml of reagent grade carbon tetrachloride by means of a graduate.
Reserve two of the flasks for the blank determination.
Into each of the other flasks introduce 14 to 16 gm of the sample weighed to the nearest 0.01 gm and swirl to effect complete solution.
Into each flask pipet 25 ml of the Wijs solution using pressure rather than suction to fill the pipet and again swirl to effect solution.
Allow the flasks to stand in the dark for 30 minutes at room temperature.
To each flask add 20 ml of the 15 percent potassium iodide solution, and 100 ml of distilled water.
Titrate immediately with standard 0.1 N sodium thiosulfate until the yellow color almost disappears. Add two ml of the starch indicator solution and continue titrating to the disappearance of the blue color. If the sample titration is less than 50 percent of the blank, repeat the determination using a smaller size sample.
Calculation:

$$\frac{(B - A)N \times 12.69}{\text{gm sample}} = \text{total unsaturation as iodine number}$$

A = ml of N normal $Na_2S_2O_3$ required for the sample

B = average ml of N normal $Na_2S_2O_3$ required for the blank

Suspended matter:
Invert a bottle of the sample and examine by transmitted light.

SAMPLE APPROVAL:
The supplier shall forward to the laboratory of the purchaser at the point of delivery a one pint sample taken from each manufacturing batch of this material scheduled for shipment to the General Electric Company or others designated by the General Electric Company. This sample shall be designated as "Advance Sample, Lot No. ____, O-E Material ____."
Approval of this advance sample by the General Electric Company does not constitute approval of the shipment. General Electric Company approval will be based upon the material as received at its destination and will be based not only on conformity to the properties specified herein but on conformity to all performance characteristics deemed essential by the purchaser.

MONS 034842

(Continued on page 3)

CERTIFICATE OF TEST:

The vendor shall submit promptly to the laboratory of the purchaser at the point of delivery a certificate of test in duplicate so that it will be received on or before receipt of shipment. The certificate of test shall include the following information:

Specific Gravity 20/20 C
Acid number, %, max
Color, max
Epoxide content, min, %
Iodine number, max
Viscosity at 25 C, centistokes, max
Suspended matter

This certificate shall also contain the G-E designation, purchase order number, tank car number, lot number, and date shipped, so that the certificate may be identified with the shipment.

PACKING AND MARKING:

Material shall be shipped in tank cars or drums, as specified on the purchase order.

All containers shall conform to ICC regulations and shall be of a type having no deleterious effect on the material. Extreme care shall be exercised by the supplier in the selection and use of common carrier containers or containers provided by the supplier. Such containers shall be carefully inspected by the supplier to assure that they are free of injurious foreign matter.

Adequate precautions shall be taken by the supplier to assure retention of the properties and characteristics of the material, as specified herein, upon arrival of the shipment at point of delivery.

Each container shall be legibly marked with the purchase order number, the manufacturer's name, the G-E designation and the lot number.

MONS 034843

GENERAL ELECTRIC

CAPACITOR DEPARTMENT
SPECIFICATION
FOR
FULLER'S EARTH

Orders and Correspondence Must Specify Comp'ny Number

D50PC12-S1

May 10, 1964

G-E Material D50PC12 identifies Fuller's Earth as follows:

D50PC12A - 80/300 mesh
D50PC12B - 60/90 mesh

PROPERTIES

	D50PC12A	D50PC12B
Moisture, %, maximum	8.0	8.0
Ignition Loss, %, maximum	18.0	18.0
Screen Analysis, %		
Retained on U.S. No. 50 mesh screen, maximum	-	5.0
Retained on U.S. No. 60 mesh screen, maximum	3.0	30.0
Retained on U.S. No. 80 mesh screen, maximum	20.0	-
Through U.S. No. 100 mesh screen, maximum	-	2.0
Retained on U.S. No. 325 mesh screen, minimum	60.0	-

MANUFACTURE

Material shall be Fuller's Earth of the attapulgus clay type, free from dirt and other contamination.

REFEREE METHODS

Sampling	ASTM D392
Moisture content	ASTM D280, Method A
Ignition loss	ASTM D50
Screen analysis (1)	ASTM D392, Method A

(1) 50 gm sample. Washed.

CERTIFICATE OF TEST

When requested, the manufacturer shall submit promptly to the purchaser at the point of delivery a certificate of test in triplicate showing that the material conforms to this specification. This certificate shall be addressed to the section, unit and person specified on the purchase order, and shall contain the G-E designation and the purchase order number so that the certificate can be identified with the shipment.

FACTORY TRIAL APPROVAL

Approval of material to this specification will be based on factory trial. Once approved, no change shall be made in the composition or properties of the approved material without the prior knowledge and consent of the purchaser.

PACKING AND MARKING

Material shall be shipped in 5-ply paper bags to meet ICC and carriers' regulations.

Each bag shall be legibly marked with the purchase order number, the manufacturer's name, the lot or batch number, the weight, and the G-E designation.

MONS 034844

RPSEBOK

PROCESS INSTRUCTIONS

GENERAL ELECTRIC

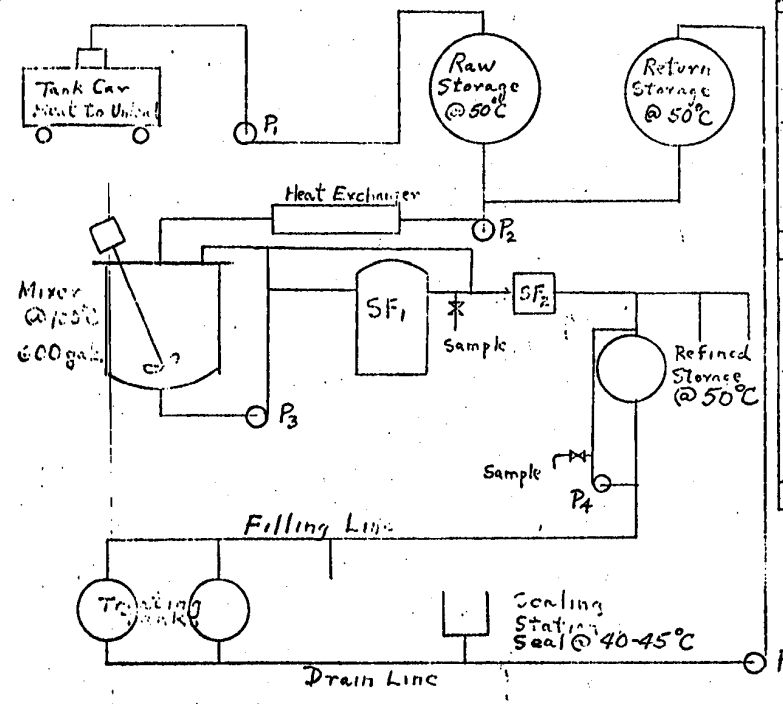
GAS 4367

REV NO. | CONT ON SHEET | SH NO.

TITLE: FLOW SHEET - POWER CAPACITOR REFINING & TREATING OPERATIONS

FIRST MADE FOR: [illegible] PYRANOL

REVISIONS



PUMP	TYPE	RATING
P1	Gear	90 g.p.m.
P2	"	35 "
P3	Centrifugal	30 "
P4	Chempump	10 "
P5	Gear	35 "

SF1 = Sparkler Filter 35-D-17

SF2 = Sparkler Filter VR12-6

PRINTS TO

[illegible] | APPROVALS | CAPACITOR DIV OR DEPT. | HUDSON FALLS LOCATION | GAS 4367 CONT ON SHEET | SH NO.

[illegible] 3, 1967

PF-150-WA' (11-61) PRINTED IN U.S.A.

CODE IDENT NO.

MONS 034846

CAPACITOR DEPARTMENT
NIAGARA FALLS, NEW YORK
APRIL 27, 1966

COPIES: A Katchman
GB Monroe

SUBJECT: Characteristics of Pyranol With and Without EP-206 Stabilizer

Mr. R.S. Dunham, Mgr.
Adv. Mfg. Process Development
O F F I C E

At your request, some tests were made to determine the changes in test characteristics of 1499 Pyranol after adding EP-206 stabilizer. Your interest in this subject comes from your proposed method of filling manifold treated units. It is possible, with available types of metering equipment, to add the epoxide (accurately measured to the desired concentration) to the Pyranol immediately before use. This would eliminate the refining step, provided that the characteristics of the impregnant were satisfactory.

To evaluate this proposal, I worked with Gerry Monroe of the Materials and Processes Laboratory. For the experiment, several tests were made on 1499 Pyranol using the test cells he normally uses on Pyranol II. When the resistivity showed that the cells were conditioned, control tests were made for power factor and resistivity. Weighed amounts of EP-206 taken from the factory storage tank were then added and the measurements were repeated. Here are the results of two tests:

SAMPLE	PF-1Ke	PF 100 cyc	Resist. x10^9
Control - No EP-206	.021	.25	8 000
Repeat - 0.35% EP-206	.10	>3%	240

Cells were then rinsed three times with good 1499 Pyranol and then filled with Pyranol.

Control - No EP-206	.077	.78	3 420
Repeat - 0.35% EP-206	.105	>3%	200

The above results were shown to Art Katchman. He remembered some recent tests that they had made in the same manner and had Tom Shea show me their results:

	PF 100 cyc	Resist. $X10^9$
1 - Control - No EP-206	1.7	13 600
2 - Repeat - 0.36% EP-206*	>6%	216

* - This EP-206 was from an old batch of Lab. distilled epoxide.

3 - Control	2.4	8 000
4 - Repeat - 0.35% EP-206**	>6%	64

** - As received EP-206

Above samples 2 and 4 were heated in an oven for (4) hours at 100°C. to see if any change occurred.

2 - With distilled EP-206	2.7	480
4 - With as received EP-206	>6%	128

Additional heating over a weekend at 100°C. produced no essential changes from above results.

In some previous work for Art Katchman, a batch of Pyranol II was prepared in the Pilot Shop refining system. Some interesting results on this experiment were reported in my Report MF 65-101 dated October 12, 1965. At that time, we refined 1499 Pyranol to a resistivity of 4600 X 10^9 with a 100 cycle power factor of 0.47%. Freshly distilled epoxide supplied by Katchman was then added directly to the batch without going through the filter bed. The resistivity dropped to 2880 X 10^9 and the power factor was 0.46%. A couple hours of refining through the filter press actually dropped the resistivity to 1960 X 10^9 and the power factor rose to 0.54%.

As you can see, there are some wide variables in the data. In the case of the Pilot Shop material, there was a negligible drop in resistivity but the other two experiments show decreases of more than a decade in resistivity. You will probably want to explore this further with the lab. people before deciding to proceed on a metering type of fill.

G.A. Smith
Adv. Mfg. Process Development
Bldg. 10-2

bjc

c. After Treate

Hudson Falls - The treat operator will secure in bottles supplied by the Treat Control Laboratory one pint samples of Pyranol from each after treat liner and label as to treat tank, liner treat number, and P.I. of the treat process. These samples will be held until the subject treat or liner unit samples have been fully tested and passed.

Fort Edward - The Laboratory technician will secure one pint sample daily from both the automatic treat and the vapor treat for testing that day.

	Hudson Falls	Fort Edward
Thermal chemical chlorides, ppm, max. (Note #3)	.75	.75

#1. For any tank with a resistivity below this control limit, the following parties must be notified before release.

1 - Product Engineering
2 - Quality Control

#2. Run at Fort Edward only. Higher values indicative of treat difficulties. Material will be rejected if higher than .45.

#3. In the event that a thermal chemical chloride result is over specification an additional sample will be run and the final answer will be an average of the samples tested.

2. A13B2A - 1436 Pyranol

a. Mixer

	Method	Hudson Falls (Check once a week or more frequently on request)	Fort Edward (Check each mixer)
Refractive index @ 25°C	ASTM D901-56	1.6185-1.6215	1.6185-1.6215
B-chlor, %,		.9 - 1.1	.9 - 1.1

b. Refined storage - check each tank for:

Test	Method	Hudson Falls	Fort Edward
Resistivity, ohm-cm, min. @ 100°C	CLTM 37	600×10^9	600×10^9
Water content, ppm, max.	G.E. E4C45	20	20
Refractive index @ 25°C	ASTM D901-56	1.6185-1.6215	1.6185-1.6215
B-chlor, %		.9 - 1.1	.9 - 1.1
Aliphatics, %, max. (by Central Lab)	CLTM 14	.02	.02
Dowtherm (#2)			NDA

SUBJECT : TEST ON MINERAL OIL, PYRANOL AND CASTOR OIL

WRITTEN BY P.M. Johnson
ISSUED BY C.R. Austin

THIS INSTRUCTION IS VOID AFTER 1-16-64

Process P3C-PC23 covers all in-process tests made by the Quality Control Materials Process Laboratory Unit on A13A2 (5314 mineral oil), A13B1C (1499) and A13B2A (1436) Pyranols and A50PC7 castor oil, used in Building #1, Hudson Falls Plant and in the Fort Edward Plant.

GENERAL

Samples are taken and tests run by the Q.C. M&P factory control Laboratory representative unless otherwise indicated.

Test methods are referenced in the material specification except those listed below:

Refined treating materials must be tested and passed as indicated before they may be used for impregnating capacitors.

Out of control tanks, trends, or deviations shall be brought to the attention of the Treat Unit Manager and the responsible Quality Control personnel at once.

PROCEDURE

1. A13B1C - 1499 Pyranol

   a. Refined storage - check each tank for:

Test	Method	Control Limit Hudson Falls	Ft. Edward
Resistivity, ohm-cm, min. @ 100°C	CLTM 37	4000×10^9 (#1)	2400×10^9 (#1)
Water content, ppm, max.	G.E. E4C45	15	20
Refractive index @ 25°C	ASTM D901-56	1.6240-1.6260	1.6240-1.6260
Fluorescence		none	none
B-chlor		NDA	NDA
Thermal chemical chlorides, ppm, max. (Note #3)	CLTM 20	.75	.75

   b. Return storage - check each day that material is taken from at Hudson Falls and four times weekly at Fort Edward for:

Test	Method	Hudson Falls	Fort Edward
Water content, ppm, max.	G.E. E4C45	60	60
Refractive index @ 25°C	ASTM D-901-56	1.6240-1.6260	1.6240-1.6260
B-chlor		NDA	NDA
Fluorescence		none	none
Aliphatics, %, max.(by Central Lab)	CLTM 14	none	none
Dowtherm, max.			.45 (#2)
Thermal chemical chlorides, ppm, max. (Note #3)	CLTM 20	.75	.75

MONS 034850

THIS INSTRUCTION IS VOID AFTER 1-16-64

c. After Treat (#4)

	METHOD	HUDSON FALLS	FORT EDWARD
Refractive index @ 25°C	ASTM D901-56	1.6185-1.6215	1.6185-1.6215

#4. Manufacturing must obtain sample from each liner for test at Hudson Falls. Manufacturing must obtain sample from each available liner otherwise raw storage is checked.

3. A13A2 - 5314 Mineral Oil

a. Refined storage (#5) - check each tank for:

Resistivity, ohm-cm, min. @ 100°C	CLTM 37 G.E. E4D2	50×10^{12}	50×10^{12}
Water content, ppm, max.	G.E. E4C45	20	20
Refractive index @ 25°C	ASTM D901-56	1.4890-1.4922	1.4890-1.4922
96 hour aging at 100°C			
Resistivity, ohm-cm, min.	CLTM 37	7×10^{12}	7×10^{12}
Acidity, mg.KOH/gm, max.	CLTM 36 ASTM D974	.014	.014
Color, ASTM, max.	ASTM D901	1.5	1.5

#5. Hudson Falls - Tanks containing all new material are tested for resistivity, water content and RI before release; 96 hour aging is run, but the tank not held for the result. If the tank contains reprocessed material, it is held for all tests.

Fort Edward - Tanks are tested for resistivity, water content and RI before release; 96 hour aging is run, but the tank is not held for the result.

4. D50PCY - Castor Oil

a. Since this material will not be refined at Hudson Falls, all approved material will be used directly from the shipping drums. For this reason only after treat samples are required.

b. After Treat (#6) - check each liner for:

Acid number, mg.KOH/gm, max.	AOCS Ca5a40	not available	not applicable
Resistivity, ohm-cm, @ 100°C, min.	G.E. E4D2		
Water content, ppm, max.	G.E. E4C45		

#6. Hudson Falls - The treat operator will secure one gallon sample from each after treat liner and mark it with the treat number and the date. The sample will then be given to the Treat Laboratory operator for transmittal to the Central Q.C. M&P Laboratory, for testing and grading. The after treat castor oil will be pumped back into the original shipping drums and tagged with the treat number and the date.

MONS 034851

Process P26D-PC4 covers the procedure for refining A13B1C (1499) Pyranol at the Fort Edward plant.

REFERENCES:
P3C-PC23 Refined Pyranol lab checks
P26D-PC1 Refining of Pyranols 1436, 1476 and 1499

GENERAL:
This Process Instruction covers only refining A13B1C (1499) Pyranol at the Fort Edward plant.
Refer to P26D-PC1 for refining other Pyranols at Fort Edward, and all Pyranols at Hudson Falls.
Only A13B1C (1499) Pyranol will be refined in this refining system.
Material from the filter blow out lines may be reused. Material from other sources such as drippings from sealing benches and pumps must be scrapped.

EQUIPMENT:
Mixing tank with agitator
Raw storage tank
Refined storage tanks (3-10,000 gal. & 2-2,000 gal.)
Sparkler filter, Model 33-D-17
Sparkler filter, Model VR-12-6

MATERIAL:
A13B1C (1499) Pyranol
Fullor s Earth, 60/90 mesh dried 4 hours at 400°C or undried.

PROCEDURE:
Charging the Sparkler Presses
Prior to charging, the presses must be cleaned according to instructions.
Fill the mix tank approximately 2/3 full and start agitation
Add 150 pounds of 60/90 mesh Fuller's Earth. Use undried earth if the primary objective is to remove chlorides. Use dried earth if the primary objective is to remove water.
Agitate for approximately 1/2 hour, or until all the earth is well suspended.
Circulate the mixture through the Sparkler filter to be charged until all of the earth is removed from the mix tank.
When the charging is complete, transfer the clear Pyranol to a raw or used storage tank.

Refining A13B1C (1499) Pyranol
Throughout the storage, refining and aging cycles, A13B1C (1499) will be held at a minimum of 50°C.
When filling, recirculating or aging a refined storage tank, a vacuum of 25" of mercury or higher will be maintained.
Refined storage tanks will be filled from a raw or used storage tank. During this filling cycle, the Pyranol must pass first through the large Sparkler press (Model 33-D-17), and then through the small Sparkler press (Model VR-12-6).
To refine a tank of Pyranol, it must be continuously circulated through a Sparkler press (model 33-D-17) for 12 hours.
At the end of this time, the Process Control Laboratory will take a sample for testing.
If the Process Control Laboratory has approved the sample, the Pyranol may be used to impregnate capacitors after it has completed a 6 hour vacuum aging cycle.
A log sheet indicating date, time filled, time the aging cycle will be complete and Process Control Laboratory approval will be obtained for each tank.

Process P3B-PC2 covers the procedure for acceptance testing of tank car shipments of Pyranols A13B1 (1476) and A13B1C (1499) before handling.

OTHER PROCESSES REFERENCED:

P26D-PC1 - Refining of Pyranols
P2A-PC6 - Handling tank car shipments of Pyranol

GENERAL:

Receiving: A sample for the acceptance tests will be taken from each incoming tank car by a properly instructed technician of the M & PT Unit BEFORE UNLOADING.

When the tank car is received, it is spotted in the proper unloading areas at Hudson Falls and Fort Edward, equipped with steam, pumping and storage facilities.

As stated in Process P2A-PC6, Pyranols must be heated to proper temperatures for unloading.

Handling: For sampling purposes, it is only necessary to heat Pyranols sufficiently for the M & PT representative to obtain a sample for test purposes.

Tests must be performed and Pyranols approved by the M & PT Unit BEFORE THE MATERIAL IS UNLOADED.

Procedure: The M & PT Unit ordinarily performs the tests listed in GE Specification A13B1 for GE Materials A13B1 and A13B1C except the tests for viscosity, fire point and sulfate. These exceptions are performed only when requested by either the Product Engineer Manager or the Laboratory Manager.

The following tests are ordinarily performed on all incoming tank cars of A13B1C Pyranol (1499):

Condition	Water
Refractive Index	Dielectric Constant
Acid Number	Resistivity
Free Chlorides	Dechlorination
Corrosion Stability	*Infrared
Acid No.	
Free Chlorides	
Color	
Condition	

*This test is performed but not listed in the material specification.

The following tests are ordinarily performed on incoming tank cars of A13B1C Pyranol according to the multi-level continuous sampling plan H106:

Specific Gravity	Pour Point
Color	Flash Point
Distillation Range	Dielectric Strength

MONS 034853

The tests conducted on incoming A13B1 (1476) tank cars are the same performed on A13B1C (1499) excepting flash point. Also, a 50% distillation determination is made.

In the event that either material (A13B1 or A13B1C) fails to meet the specification limits of the required tests, a second sample must be taken from the tank car and all tests which the first sample failed must be repeated. In addition, the advance sample taken by the vendor when the tank car is loaded must be evaluated.

In the event that the only acceptance test failure is the resistivity test, the material may still be accepted providing that the resistivity is more than 50% of specification limit. Experience has shown that low resistivity Pyranols can be raised to our process limit of resistivity by our Fuller's Earth filtering process (P26D-PC1).

CAPACITOR DEPARTMENT PROCESS

ACCEPTANCE TESTING OF PYRANOLS - A13B1 (1476) & A13B1C (1499)

In the event the material fails to meet the specifications, the results must be reported immediately by the M & PT Unit to the responsible Product Engineering Manager and the Laboratory Manager, or their respective Supervisors whom they have designated responsible.

Product Engineering Managers will secure advice from the Laboratory Manager or his designated alternate, and will advise the Supervisor of the M & PT Unit to reject or accept the tank car in question. This Engineering decision will be recorded on the test report issued by the M & PT Unit. In addition, the M & PT Unit will issue an IR showing that the material fails to meet the required specification. The Engineering disposition will be recorded on the IR.

In the event that a tank car meets all requirements, but in the opinion of the Supervisor of the M & PT Unit exhibits unusual characteristics in any respect, the lot must be held up and referred to the responsible Engineering Manager for disposition by the procedure described in this process for cases of failure to meet specification requirements.

Tank cars which meet all specification requirements and which exhibit no unusual characteristics, will be released by the M & PT Supervisor. The results of these tests will be published quarterly advising the responsible Product Engineering Manager, Purchasing Agent, Manager of Quality Control and other interested persons by means of a copy of the report. In the event that anything is found unusual or the material fails to meet any of the specifications, the pertinent information will be published in an individual test report issued by the M & PT Unit.

MONS 034854

GENERAL ELECTRIC CAP 894

SUPERSEDES ISSUE OF June 9, 1958

CAPACITOR DEPARTMENT
PROCESS Instruction

INSTR. NO. P26D-PC5
PAGE 1 OF 2
WRITTEN BY G.A. Smith
ISSUED BY E.S. Dunham

SUBJECT
REFINING OF PYRANOL II AT FORT EDWARD PLANT

THIS INSTRUCTION IS VOID AFTER December 4, 1967

Process P26D-PC5, is an outline of the procedure for refining D50PC5A Pyranol made which has been stabilized with D50P132-S1 material.

REFERENCES:

P3C-PC23	Refined Pyranol Lab Checks
P3C-PC40	In-Process Testing Pyranol II
P25A-PC1	Operation of Pyranol Resistivity Bridges
P2A-PC10	Handling of Epoxide

GENERAL:

This Process Instruction describes the refining of stabilized 1499 Pyranol (Pyranol II). Only Pyranol II will be refined in this system.

EQUIPMENT:

Mixing tank and agitator
Raw and refined storage tanks
Sparkler Filter, Model 33D-16
Sparkler Filter, Model VR-12-3
Epoxide Storage Tank
Transfer Pump
Proportioning Pump

MATERIAL:

A13B1C (1499)
D50P132-S1 Unox 206 Epoxide (see Material Approval)
Fuller's Earth, 60/90 mesh dried 4 hours at 400°C.

PROCEDURE:

Charging the Sparkler Presses

Prior to charging, the presses must be cleaned of the spent fuller's earth from previous runs. The mix tank is filled with 70 - 80°C Pyranol, and the agitator is started. A maximum charge of 75 pounds of 60/90 mesh fuller's earth is added to the mix tank. The slurry is agitated for one-half hour and then circulated through the large Sparkler filter. Pyranol is transferred through the filter to the return storage tank.

MONS 034855

Saturating the Fuller's Earth with Epoxide

After the press has been charged with fresh fuller's earth, it must be saturated with epoxide before it can be used for refining. This is done by circulating Pyranol II through the earth.

The epoxide and aliphatic content of the Pyranol II must be known before attempting to saturate new fuller's earth.

SUBJECT
REFINING OF PYRANOL II AT FORT EDWARD PLANT

WRITTEN BY G.A. Smith
ISSUED BY E.S. Dunham

THIS INSTRUCTION IS VOID AFTER December 4, 1967

Saturating the Fuller's Earth with Epoxide (continued)

Epoxide in the amount of 10 - 12% by weight of the fuller's earth must be added to saturate the earth. For a 75 pound batch of earth, the quantity is 7.5 to 9.0 pounds. Addition is accomplished by using the system set up for this purpose.

Circulate the return tank of known epoxide and aliphatic content through the new fuller's earth and sample every hour until three (3) successive measurements indicate no change in alophatic and epoxide content.

Operating the System

All returns for any given day must be back in return storage before testing. The return storage tank is constantly circulated through the circulation pump while returns are being made.

After all returns are in the storage tank, take a sample and test for epoxide concentration. Add necessary make-up 1499 Pyranol from raw storage. Add amount of epoxide specified by Process Control. Circulate new blend for a minimum of two hours.

Sample and test for epoxide and aliphatic concentrations specified in P3C-PC40. Continue circulation while tests are being made. When tests are okay, transfer Pyranol II through filter press and cone to refined storage tank. After transferring, sample will be taken from refined tank. Continue circulation through filter and cone until Pyranol II is approved by Process Control by written autogram.

Place refined tank on aging for a minimum of 4 hours before use.

SAFETY:

Special care should be taken when handling Unox 206 stabilizer. Potential hazards from this material are described in Process Instruction P2A-PC10.

Material Approval

The Epoxide used in the preparation of Pyranol II must be certified in writing by the Laboratory prior to use.

MONS 034856

CAPACITOR DEPARTMENT
PROCESS Instruction
SUBJECT IN-PROCESS TEST BY MATERIALS TESTING AND ANALYSIS UNIT ON PYRANOL II - HUDSON FALLS & FORT EDWARD

6/4/66 INSTR. NO. P3C-PC40
PAGE 1 OF 2
WRITTEN BY G. A. Smith
ISSUED BY E. S. Dunham

THIS INSTRUCTION IS VOID AFTER .....June 1, 1969

Process P3C-PC40 covers all in-process tests made by the Materials Testing & Analysis Unit on A50PC8A and A50PC8B used in the Fort Edward Plant and Hudson Falls Plant.

GENERAL

Samples are taken and tests run by the Materials Testing & Analysis Unit representative unless otherwise indicated.

Test methods are referenced in the material specification.

Refined treating materials must be tested and passed as indicated before they may be used for impregnating capacitors.

Out of control tanks, trends, or deviations will be published in the monthly process control report.

G. E. CLASS IV
NOTICE
The information contained herein is proprietary to the interest of the General Electric Company only.

PROCEDURES

1. Refined Storage - check each tank for:

TEST	A50PC8A CONTROL LIMIT FORT EDWARD	A50PC8B CONTROL LIMIT HUDSON FALLS
Aliphatic Content, Infra-red Analysis, %, Max.	See Note 1	See Note 1
Resistivity, ohm-cm, Min.	1100×10^9	2000×10^9
Epoxide Content, %	0.35 ± 0.05	0.35 ± .05
Fluorescence	NDA	NDA
Power Factor, %, 100°C, 100 cycles (Max)	---	0.75

2. Return Storage - check each day material is added to or taken from tanks for:

Fluorescence	NDA	NDA
Epoxide Content, %	0.35 ± 0.05	0.35 ± 0.05
Aliphatic Content, Infra-red Analysis	See Note 2	See Note 2

3A. After Treat - Round & Rectangular Tanks: The Laboratory will supply clean pint sample bottles to Manufacturing for after treat samples. Manufacturing will secure one pint samples of Pyranol from each after treat and label as to treat tanks, run number and type of treat, and turn them over to the Laboratory. After treat samples of Pyranol II will be tested on a reduced sampling plan or as required by problems encountered for the following:

Aliphatic Content, Infra-red Analysis	See Note 2	See Note 2
Epoxide Content, Min.	0.25	0.25
Fluorescence	NDA	NDA

3B. After Treat-Automatic Treat: The Laboratory will supply clean pint sample bottles to Manufacturing for after treat samples. Manufacturing will secure one pint samples of Pyranol from return storage tank at completion of each run of the automatic treat and label as to treat, run number, and type of treat and turn them over to the Laboratory. After treat samples of A50PC8A will be tested as described in 3A.

MONS 034857

THIS INSTRUCTION IS VOID AFTER June 1, 1969

PROCEDURES (Continued)

NOTE 1: The aliphatic content shall be held to less than .0% above the active epoxide concentrate for both Fort Edward and Hudson Falls.

NOTE 2: The aliphatic content of the after treat sample shall not exceed that of the pre-treat (Refined Storage) sample.

These tests are conducted to determine the effect that treat has had on the A50PC8A (Pyranol II), and to establish the quality level for after treat material being returned to storage.

MONS 034858

CAPACITOR DEPARTMENT
Process Instruction

INSTR. NO. P2A-PC10
PAGE 1 OF

SUBJECT : SAFETY RULES FOR HANDLING AND USE OF PYRANOL II STABILIZER (EPOXIDE 206)

WRITTEN BY C.A. Smith
ISSUED BY E.S. Dunham

THIS INSTRUCTION IS VOID AFTER 1/11/63

General

Process P2A-PC10 covers the procedure for the handling and storage of Unox Epoxide 206.

Reference

Union Carbide Corporation Bulletins
F-40606 Nov. 1959 and F-40132D June 1960 and P2B-PC3

Safety & P2B-PC3:

Epoxide resins are primary skin irritants. Prolonged and repeated contact may cause delayed and serious injury to the skin. In commercial practice, precautions which permit only intermittent skin contacts with these materials are effective in preventing the occurrence of dermatitis among normal workers for a protracted period of time. However, under these circumstances, a small number of hypersensitive workers have been observed to develop a sufficient degree of sensitivity, to be affected adversely by these materials. Unfortunately, such individuals cannot be identified in any group of workers until recurrent dermatitis is displayed. Because of this combination of circumstances, it is necessary for all workers to use protective measures to prevent all contact of the skin with the epoxides.

If these materials come into contact with the skin, they should be washed off immediately with soap and water or removed with a waterless skin cleaner. Clothing which is soiled or wet with these materials must be removed and not worn again until it has been thoroughly washed.

Avoid inhalation of vapors or mists even though the hazard by inhalation is slight. The eyes are moderately harmed by the undiluted fluid. Eye protection is required when handling the chemical. In case of eye contact, the eye should be given a 15 minute emergency washing in the dispensary, and a physician should see those cases in which discomfort persists.

Persons who are working directly with epoxides must wash hands before eating, drinking or smoking.

Eating, drinking or smoking in working area where epoxides are used or prepared, is not permitted.

As shipped, Unox 206 is completely stable at room temperature for periods exceeding one year. Contact with acids, alkalies, water or water vapor may cause partial condensation. Other strong catalytic agents such as Lewis acids or other acid or basic salts may cause rapid polymerization and the rapid evolution of heat. Therefore, Epoxide 206 should not be stored in any area where it may contact any of the above materials. Any partially used drums or cans of this material should be purged with dry Nitrogen before storage.

Adequate ventilation must be provided in the mealing area to minimize [illegible] hazards existing when Pyranol II, containing a very small percentage of stabilizer, is subjected to elevated temperatures.

O.C
INSTRUCTIONS

MONS 034860

DRAFT SPECIFICATION

E50 PC-22

DIRECT TITRATION METHOD FOR THE DETERMINATION OF EPOXIDE SCAVENGER IN 1492 PYRANOL

Apparatus:

Karl Fischer type burette equipped with drying tubes filled with Drierite or other suitable dessicant. Titration must be performed in a closed system to avoid loss of HBr. A rubber cap similar to Fischer #14-127-5 or a vented, one-hole rubber stopper can be used to attach the burette tip to the titration flask.

125 ml erlenmeyer flasks
Magnetic stirrer
Teflon-coated magnet bar

Reagents:

Glacial acetic acid, reagent grade
Chlorobenzene, Eastman or Fischer
Crystal violet indicator solution, 0.1% in glacial acetic acid
Hydrogen Bromide, anhydrous, Matheson Company

Prepare 0.1 N HBr in glacial acetic acid by bubbling HBr slowly through glacial acetic acid for a weight gain of 8.1 g of HBr/liter of glacial acetic acid.
Sodium Carbonate, anhydrous reagent grade. Dry at 120°C for at least three hours before using - store in dessicator.

Standardization:

Standardize HBr solution against 0.1 g of sodium carbonate dissolved in 10 ml of glacial acetic acid. Use five drops of indicator solution and titrate to a bluish green end point while stirring rapidly with magnetic stirrer. Titration should be performed in a closed system.

$$\text{Normality} = \frac{\text{Weight of } Na_2CO_3}{0.053 \times \text{ml of HBr}}$$

Procedure:

Weigh approximately 35 gms of Pyranol into 125 ml flask. Add five drops of crystal violet indicator solution, 10 ml Chlorobenzene, and stirring bar. Titrate with rapid stirring in a closed system to blue green end point.

Calculation:

$$\text{Weight\% of active epoxide} = \frac{100\ (\text{ml of HBr})\ (\text{Normality})\ (\text{Equiv. Wt. of Epoxide})}{(1000)\ \ (\text{Wt. of Pyranol Sample})}$$

Equivalent wt. of Epoxide 206 = 70
Equivalent wt. of Epoxide 269 = 82

HBr solution require frequent standardization - daily for most accurate results. Exclusion of light may retard loss of strength. Drying tubes filled with indicating Drierite or other suitable absorbent should be used on reservoir and burette to exclude atmospheric moisture.

This method is also applicable for the determination of epoxide purity of incoming Epoxide 206. Use approximately 0.1 gram of epoxide weighed accurately to the fourth decical place - dissolve in 10 ml of Chlorobenzene and follow method as outlined.

MONS 034861

GENERAL ELECTRIC

E50PC28-61

CAPACITOR DEPARTMENT
TEST METHOD
46K
FOR
4A,4J,4K,20A,20J,20K,30A,30J,30K,50
ALIPHATIC CONTENT IN PYRANOL II (A50PC8)

December 1, 1961

I. SCOPE:

This procedure determines the quantity, in % by weight, of "Aliphatics" in Pyranol II. The "Aliphatics" are the expression of the Infrared Absorption in the 3-4 micron ($2925\ cm^{-1}$) region of the spectra and are related to the CH_{C-H} stretching frequency.

II. APPARATUS:

The following methods and procedures have been established with the Perkin-Elmer Model 21 infrared Spectrophotometer which has been modified by the addition of Optical Scale Expension.

1. The Infrared Spectrophotometer requires Optical Scale Expansion.
2. Sample cells to match the optics. The cells are to be a matched pair of 0.100 mm spacing.
3. Syringes and necessary equipment to load and clean said cells.

III. CALIBRATION:

Quantitative Infrared Spectro Analysis requires that precise standards be established to cover the range of analysis.

For the range of this analysis standards of 0.1, 0.2, 0.3, 0.4 and 0.5% by weight of as received Epoxide 206 in 1499 Pyranol should be prepared. The weights of Epoxide 206 and 1499 Pyranol are to be determined to the nearest 0.1 mg for the calculation of percentages.

IV. TEST SAMPLES:

The samples should be taken, handled and stored in compliance with standard practices for purity preservation.

V. PROCEDURE:

(Specific for the modified Model 21 Perkin-Elmer Infrared Spectrophotometer).

All analysis shall be of the differential type, that is, versus a standard lot of 1499 Pyranol.

A. Instrument Conditions

1. Spectra Range - 3.0 - 4.0 microns
2. Source Intensity - Maximum -) 0.3 amps.
3. Optical Scale Expansion - 5X
4. Slit Controls
   a) Auto
   b) Filter - out
   c) Resolution - 10.00
5. Amplifier Controls
   a) Test signals - off
   b) Gain - 4.5 - 5.0 nominal
   c) Response - 4
6. Scanning Control
   a) Speed - 15 minutes
   b) Automatic Drum Control
   c) Automatic Suppression - 0

B. Sample Cell

Extreme care must be taken to insure that the sample and reference cells be free from all contamination.

The cleaning of the reference cell, 1499 Pyranol, can be accomplished by a thorough washing with petroleum ether but the sample cell, Pyranol II, must first be rinsed with 10 ml of benzene then washed with the petroleum ether.

VI. INTERPRETATION OF SPECTRA:

By drawing a base line across the top of the absorption spectra the depth of the absorption curve is measured perpendicular from the maximum absorption point to the base line. Expressing this in percent transmittance a direct comparison can be made to the calibrated standards.

GENERAL ELECTRIC

TEST METHOD

E4C48-S2

UNSTABLE CHLORINE COMPOUNDS IN ASKARELS

Supersedes E4C48-S1

G-E Test Method E4C48 governs the determination of the presence of unstable chlorine compounds in chlorinated biphenyls (askarels).

PRINCIPLE OF METHOD:

This method is based upon the hydrolysis of unstable chlorine compounds in askarels by methanolic sodium hydroxide. The resulting chloride ion is determined potentiometrically by titration with silver nitrate solution in an essentially nonaqueous medium. The measured chloride ion, reported as parts per million in the askarel sample, is indicative of the relative stability of the askarel in a dielectric system.

APPARATUS:

Beaker - 200 ml tall form (Berzelius type)

Magnetic stirrer - Fisher Scientific Co, Catalog No. 14-511-1, or equivalent with ring stand base and built-in rheostat. Set rheostat at full speed and operate through a variac to adjust its speed to prevent heating of the stirrer during the stirring operation.

Magnetic stirring bar, Teflon - One piece molded construction, cylindrical in shape, one inch long, Fisher Scientific Co, Catalog No. 9-311-9 or equivalent.

Microburet - Graduated in 0.01 ml divisions. Suitable buret may be obtained from Scientific Glass Apparatus Co, Catalog No. JM-570.

Silver electrode - Beckman silver billet electrode, Catalog No. 39261, preferred.

Glass electrode - Standard glass electrode such as Beckman electrode, Catalog No. 40498.

pH meter suitable for use with glass electrode - Model GS Beckman pH meter preferred because instrument has expanded scale and provides greatest sensitivity to incremental emf changes. Somewhat less sensitive meter such as Beckman "Zeromatic" or Leeds Northrup line operated pH meter may be used.

Water bath - Use an individual glass water bath 150 mm in diameter, 75 mm high and containing 600 ml of water heated to 40 C ± 1 C. Corning Glass Co, Catalog No. 31401 or equivalent.

Pipet, 25 ml.

Buret or pipet, graduated to deliver 0.5 ml.

Wash bottles for pure acetone, methanol, and water.

REAGENTS:

Methanol (chloride free) - Reflux 5 liters of methanol with 0.5 gram analytical reagent grade silver nitrate for 1/2 hour. Distill the methanol from the silver nitrate, discarding the first 100 ml to flush the apparatus. Distill 90% of the charge from the flask and discard the contents remaining in the flask. Chloride ion concentration should be less than 0.01 ml of 0.005N silver nitrate per 100 ml of methanol.

Sodium hydroxide reagent, 0.1 N, methanolic - Dissolve 4.0 grams of analytical reagent grade sodium hydroxide in one liter of chloride free methanol.

Sulfuric acid, 50-50 by volume - Dilute analytical reagent grade concentrated sulfuric acid with chloride-free (deionized or distilled) water. Precaution - Always pour acid into the water with constant stirring to prevent any dangerous build up of heat.

Standard silver nitrate solution, 0.005N and 0.0025N - Prepare by diluting ampoule of concentrated aqueous silver nitrate available from Anachemica Chemical Ltd, Champlain, New York. 0.005N silver nitrate may also be prepared by dissolving 0.8495 gram of analytical reagent grade silver nitrate crystals in one liter of chloride-free water. Standardize against a pure chloride standard. A sodium chloride crystal such as used in infrared spectrometer cells is a good source of pure sodium chloride. Check silver nitrate solutions at least monthly to assure a consistent reagent.

Acetone (chloride free) - Distill from silver nitrate as described for methanol. Also check by potentiometric titration to assure optimum purity. Normally a chloride content of less than 0.01 ml of 0.0025N silver nitrate per 100 ml is derived by this method.

Benzene - Use an analytical reagent grade which is normally chloride free. Check by potentiometric titration.

PRECAUTIONS:

Exercise usual analytical precautions to prevent cross contamination from other sources of halogen in the laboratory. All glassware, apparatus, and the area in which this test is run should be analytically clean.

(Continued on page 2)

MONS 034863

GENERAL ELECTRIC

TEST METHOD

UNSTABLE CHLORINE COMPOUNDS IN ASKAREL

PROCEDURE:

Weigh 25 grams of askarel into a tared 200 ml beaker to the nearest 0.01 gram on a suitable balance.

Add the magnetic stirring bar to the beaker containing the sample without the bar touching the hands.

Note: When testing the more viscous askarels, add 5 ml of benzene immediately after the stirring bar. Heat the sample until dissolved, cool to room temperature and then proceed as prescribed. Add the benzene to the reagent blank also.

Add 25 ml of 0.1N methanolic sodium hydroxide by means of a 25 ml pipet and cover the beaker with a watch glass.

Place the sample in the 40 ± 1 C water bath to a depth of 1 1/4 inches on a magnetic stirrer and clamp securely to a firm support. Stir the sample at as fast a speed as possible, without pronounced splashing, for one hour. (Alternatively, heat the sample to 40 C in a water bath, remove the sample and stir as above on a magnetic stirrer.

After the one hour stir, remove the sample beaker from the bath and add 0.5 ml of dilute sulfuric acid by means of a suitable pipet or buret. Add 125 ml of chloride-free acetone from a graduated cylinder.

Titrate the sample with 0.005N silver nitrate solution using the silver-glass electrode system. Normal samples of askarel require extremely small amounts of silver nitrate. Therefore, run the titration using 0.01 ml additions and allow sufficient time for equilibrium to be established before recording the emf change. If a change of less than 1 mv per 0.01 ml addition is observed for three or four 0.01 ml increments, use larger additions of silver nitrate such as 0.05 ml until such a change is observed. Then reduce the additions to 0.01 ml again to complete the titration. The endpoint normally is defined by two 50 mv changes. A normal titration would yield the following typical data:

MV	dMV(1)	ML	dML	dMV/dML
368	0	.06	0	0
360	8	.07	.01	8
352	8	.08	.01	8
341	11	.09	.01	11
321	20	.10	.01	20
271	50	.11	.01	50
221	50	.12	.01	50
201	20	.13	.01	20
185	16	.14	.01	16

(1) Using the Beckman GS pH meter the change is measured in 0.2 mv units and hence the meter changes observed would be five times this value (i.e. 25 units for 5 mv).

To calculate the change per 0.01 ml observed divide the mv change by the volume of silver nitrate. By plotting dmv/dml vs ml, determine the endpoint to the nearest 0.001 ml. (This gives a sensitivity of ± 0.007 ppm. To define the endpoint to ± 0.01 ml, no plotting is necessary and a sensitivity of ± 0.07 ppm is assumed.)

Run a reagent blank exactly as above omitting the askarel sample.

CALCULATIONS:

Subtract the reagent blank from the total volume of silver nitrate and for the sample, then:

$$\text{Reactive chlorine (ppm)} = \frac{\text{Net vol } AgNO_3 \times \text{normality of } AgNO_3 \times 35.46 \times 1000}{\text{Weight}}$$

PRECISION:

A rapid titration can be made to the nearest 0.1 ml using the normal potential at the equivalence point or use can be made of an automatic titrator for routine control procedures. The sensitivity in either case should be within ± 0.2 ppm of the value obtained by more refined techniques.

REPORT:

Report shall include the G-E designation of the material tested, the manufacturer's name, the purchase order number and the parts per million of reactive chlorine dechlorinated.

MONS 034864

TEST METHOD

CONTAMINATION OF CAPACITOR LIQUIDS

Supersedes E4C44-S3

G-E Test Method E4C44 governs the determination of the contaminating effect of various materials on capacitor dielectric liquids.

The following Test Methods are referenced in this method:

G-E E4C41C - Chloride Content of Solutions
ASTM D974 - Neutralization value by Color-Indicator Titration
G-E E4D2 - Resistivity of Electrical Insulating Liquids

PRINCIPLE OF METHOD:

A sample of definite surface area or size is aged in the liquid for 96 hours at 100 C and the resistivity or specific conductance determined as a measure of the contaminating effect of the sample on the liquid. Other tests such as acidity and free chlorides may also be run as warranted. The material under test is visually examined for physical change.

PRECAUTIONS:

Containers must be tightly covered to prevent cross contamination. Utmost care must be taken to prevent accidental contamination of the liquid, the sample under test or the equipment.

APPARATUS:

Circulating air oven capable of maintaining a temperature of 100 ± 2 C
800 ml beakers
Aluminum foil

REAGENTS:

Capacitor dielectric liquid specified for the test.

SAMPLING:

Samples shall be selected depending upon the material to be tested and unless otherwise specified shall be the following size:

Material	Sample size
Solids	1.0 sq inch of total surface area
Material soluble in the test liquid	0.5% by weight
Powdered or granular material insoluble in the test liquid	1.0% by weight

In handling the specimens take great care that hands do not touch the area to be tested.

PROCEDURE:

Dry sample at 150 ± 2 C for a minimum of 16 hours.
Wrap an 800 ml beaker with aluminum foil. Add 500 ml of the specified dielectric liquid and place the sample into the beaker. (Note: The liquid must not have a water content exceeding 20 ppm and should have good electrical characteristics.)
Cover the top of the beaker with aluminum foil and age the sample for 96 hours at 100 ± 2 C. Age a blank consisting of a similar beaker of the same liquid but without a sample.
At the end of the aging period, examine the test liquid and the sample for any changes in physical condition and measure the resistivity in accordance with G-E Test Method E4D2. If required, then determine the acidity of the liquid in accordance with ASTM D974 and the chloride content in accordance with G-E E4C41C.

CALCULATIONS:

Calculate percent change in resistivity as follows:

$$\text{Percent change} = \frac{100\ (\text{Resistivity of blank} - \text{Resistivity of liquid \& sample})}{\text{Resistivity of blank}}$$

Calculate the specific conductance due to contamination as follows:

$G_s = G_m - G_o$

Where G_s = Conductance due to contamination,
G_m = Measured conductance (reciprocal of resistance) of liquid which contained sample, &
G_o = Measured conductance (reciprocal of resistance) of blank

Specific conductance due to contamination = $G_s \times K$

Where K = cell constant

REPORT:

The report shall include the G-E designation of the material tested, the manufacturer's name or tradename, the purchase order number, the percent change in resistivity or the specific conductance and if determined, the acidity and chloride content of the liquid.

GENERAL ELECTRIC

E4D2-S2

TEST METHOD

RESISTIVITY OF ELECTRICAL INSULATING LIQUIDS

Supersedes E4D2-S1

G-E Test Method E4D2 governs the procedure for measuring the resistivity of electrical insulating liquids.

PRINCIPLE OF METHOD:

The volume resistance of the liquid is determined between two concentric cylindrical electrodes and the resistivity calculated.

PRECAUTIONS:

Beaker, glass plate and electrodes must be thoroughly cleaned to assure correct readings. Do not use abrasive cleaners.

APPARATUS:

Megohm meter capable of measuring at least 100,000 megohms, such as Electronic Instruments Ltd 20,000,000 megohm meter or General Radio 544B.

Capacitance bridge capable of measuring capacities in the order of 50-100 uuf.

Electrode assembly as shown in figure 1, as follows:

Concentric cylindrical electrodes made of polished nickel-plated brass, 3 1/2 inches high and with three feet 1/4 inch high equally spaced around the cylinder. The inner electrode shall have an OD of approximately 2.810 inch and the outer electrode shall have an ID of approximately 3.015 inch so that there will be a gap of 0.10 inch between them.

Pyrex glass plate 3 1/2 inch in diameter with two concentric grooves 1/32 inch deep to position the cylindrical electrodes.

800 ml Griffin type beaker.

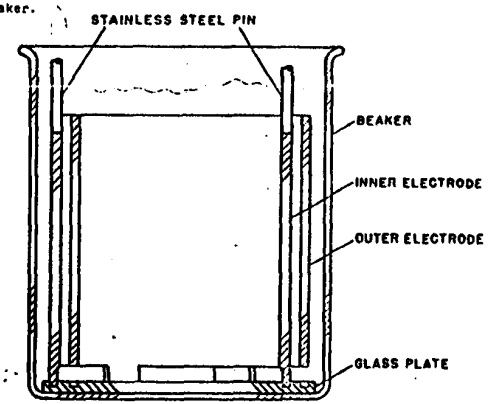


Figure 1

REAGENTS:

Acetone solvent (G-E Material D5B23B)

Alconox, trisodium phosphate, or other suitable detergent.

MONS 034866

DETERMINATION OF CELL CONSTANT:

Clean beaker, glass plate and electrodes as specified under Procedure.

Place the glass plate and electrodes in the beaker, positioning the electrodes carefully in the plate grooves so that there is a gap of 0.10 inch between them.

Measure the air capacity of the cell using the center electrode as the high terminal.

Calculate the cell constant, as follows:

$$\text{Cell constant (K)} = \frac{\text{Air capacity in uuf}}{0.0884}$$

The cell constant should be determined at least once a week, and oftener for more precise measurements. Typical values for air capacity are 67 to 72 uuf.

(Continued on page 2)

GENERAL ELECTRIC

TEST METHOD

RESISTIVITY OF ELECTRICAL INSULATING LIQUIDS

OCEDURE:

Clean the beaker, glass plate and electrodes in a trichloroethylene vapor degreaser and then wash with acetone solvent. Wash with a solution of Alconox (approximately 2%) or other suitable detergent in water until water will wet the entire surface uniformly. Wash thoroughly with distilled water and dry at 105-125 C. Care should be taken to clean the top and bottom edges of the electrodes as well as the larger surfaces.

Wrap the sides and bottom of the beaker in aluminum foil to shield it from light. Place the glass plate and electrodes in the beaker and rinse twice with fresh portions of the liquid to be tested at 100 C. Position the electrodes in the grooves of the glass plate so that there is a gap of 0.10 inch between them.

Fill the beaker with the sample and maintain at a temperature of 100 ± 0.5 C in a constant temperature bath or have the sample slightly above 100 C and make resistivity measurement when the sample cools to 100 C.

Measure the resistance of the sample at 500 volts d-c after one minute electrification using the center electrode as the high terminal.

If the resistivity of the liquid is above specifications, the cell may be stored in the liquid until the next measurement without cleaning.

CALCULATION:

Resistivity in ohm-cm = resistance in ohms x cell constant (k).

REPORT:

The report should include the G-E designation of the material tested, the manufacturer's name, the purchase order number, the resistivity and the temperature at which it was measured.

MONS 034867

CLTM-1
Date Issued: Oct. 1, 1959
Revised:

Tentative O.E. Free Chloride Test Method

(1) For normal Pyranol 100g of Pyranol is dissolved in 100 ml of acetone. The Pyranol is weighed accurately to .01 gm in a 250 ml beaker.

The acetone used here is ACS grade acetone. It normally contains about 0.01 ppm or less chloride. If the solvent blank is high, it may be purified by refluxing with $AgNO_3$ and distilling off the acetone. Other solvents of similar purity may be used, if sample solubility necessitates (i.e. Dioxane, Dimethylformamide, Glacial Acetic Acid etc.).

(2) 2 ml of 1% HNO_3 is added to the acetone-Pyranol mixture and it is stirred 5 minutes to insure proper mixing.

The nitric acid used is made from concentrated nitric acid, diluted to give a solution of 1% strength with chloride free water (distilled or deionized).

(3) The resulting mixture (from 2) is titrated with 0.0025N $AgNO_3$ using a silver glass electrode system and a Model "G" or "GS" Beckman ph meter.

The glass electrode used is a Beckman glass electrode Cat. #40498. The silver electrode is a Beckman silver billet electrode Cat. #39261. Normal samples of Pyranol require small amounts of the $AgNO_3$ reagent. For this reason 0.0025N $AgNO_3$ is used with a 1 ml automatic buret graduated to 0.01 ml. (Such a buret may be obtained from Scientific Glass Apparatus Co., Inc.) Additions of 0.01 ml of the $AgNO_3$ reagent are made allowing 1 minute between additions for proper mixing before making the emf readings. If a change of less than 1 mv is observed for an addition, larger additions are made - for instance 0.05 ml until a change of 1 mv/per .01 ml of solution is observed. When a change of this size is observed, the increment is immediately decreased to 0.01 ml of $AgNO_3$ again. The endpoint for this determination will ordinarily give a change of 40-50 mv/.01 ml addition. This change is calculated continuously during the titration from the volume of $AgNO_3$ added and emf changes observed.

To calculate the change per 0.01 ml observed, the millivolt change observed is divided by the volume of $AgNO_3$ added. For instance, if a change of 10 mv is observed for 0.02 ml addition, the change of emf/.01 ml would be 5 mv/.01 ml and the next increment should be adjusted downward to 0.01 ml to avoid missing the endpoint. The information would be recorded as follows:

MV	Δ MV	ML	Δ ML	Δ MV/Δ ML
1000	- -	0.00	- -	0
990	10	.02	.02	5
920	10	.03	.01	10
890	30	.04	.01	30
840	50	.05	.01	50
790	50	.06	.01	50
760	30	.07	.01	30
700	10	.08	.01	10
745	5	.09	.01	5

By plotting ΔMV/Δml vs ml the endpoint can be found to the nearest 0.001 ml easily. A little experience with this method of chloride determination on known solutions will prove valuable and in a short time precision of .001 ml will be routine.

ethylene vapor degreaser and then wash with acetone solvent. Wash with a solution of Alconox (approximately 2%) or other suitable detergent in water until water will wet the entire surface uniformly. Rinse thoroughly with distilled water and dry at 105-125°C.

Note 1 : Care should be taken to thoroughly clean all surfaces which will come in contact with the foil or Pyranol.

Note 2 : The electrodes should be conditioned as specified in E4D2.

Stack sufficient layers of foil to make a weight of approximately 50 grams plus an outer wrap. Holding this pad with forceps, discard the top and bottom layers and tie a cord around the stack to make a bundle. Weigh this bundle, cutting off pieces until a 50 ± 0.1 gram weight is obtained.

Note 3: Extreme care should be taken to protect the sample from any source of contamination, including handling.

Dry the specimen at 150 ± 2°C for a minimum period of 16 hours.

Wrap a beaker with aluminum foil to shield it from light. Add 500 ml. of A13B1C and heat to 100 ± 2°C.

Note 4 : Experience has shown that room temperature Pyranol will reach 100°C in approximately an hour when placed in an oven.

Note 5 : The Pyranol used must have a minimum resistivity of 1000×10^9 ohm-cm and a water content not exceeding 20 PPM before heating.

Place the beaker containing the hot Pyranol in the oven containing the dried sample. Grasping the sample by the string, hold it over the Pyranol and cut the string so that only the foil sample falls into the hot Pyranol. Discard the string.

Replace the aluminum foil cover and age the sample for 96 hours at 100 ± 2°C. Age a blank consisting of a similar beaker of the same Pyranol without a sample.

At the end of the aging period, pour the Pyranol into a beaker containing the conditional electrode assembly. Measure the resistivity in accordance with E4D2.

Next, determine the acidity of the Pyranol in accordance with ASTM D974 and the chloride content in accordance with ASTM D878.

CALCULATIONS:

Calculate the specific conductance as follows:

$$G_s = G_m - G_o$$

where G_m = measured conductance (reciprocal resistance) of insulating Pyranol which contained the sample.

G_o = measured conductance (reciprocal resistance) of control sample.

G_s = conductance due to contamination.

then $G_s \times K$ = specific conductance due to contamination.

K = cell constant.

REPORT:

The report shall include the complete identification of the material tested,

type of cell used, the specific conductance, the acidity and chloride content of the insulating Pyranol after test.

RAL

R.A. Larrick
9/23/59

CLTM-5
Date Issued: October 1959
Revised:

This test method governs the determination of the contamination of GE Material A13B1C (1499) Pyranol by capacitor paper.

PRINCIPLE OF METHOD:

A sample is aged in Pyranol for 96 hours at 100°C and the change in resistivity determined as a measure of the contaminating effect of the paper sample on the Pyranol.

APPARATUS:

Circulating air oven capable of maintaining temperature of 150 and 100 ± 2°C
600 ml. beakers
Megohm meter capable of measuring at least 100,000 megohms, such as, Electronic Instrument, Ltd. 20,000,000 megohm meter.
Analytical balance
Forceps
Scissors
Aluminum foil
Electrode assembly consisting of concentric cylindrical electrodes made of polished nickel-plated brass, 3-1/2 inches high and with three feet 1/4 inch high equally spaced around the cylinder. The inner electrode shall have an O.D. of approximately 2.81 inch and the outer electrode shall have an I.D. of approximately 3.015 inch so that there will be a gap of 0.10 inch between them.
Pyrex glass plate 3-1/2 inch in diameter with two concentric grooves 1/32 inch deep to position the cylindrical electrodes.
Twine

REAGENTS:

1499 Pyranol, GE Material A13B1C
Acetone solvent, GE Material D5B32B
Alconox, tri sodium phosphate or other suitable detergent
Distilled water

PRECAUTIONS:

Beakers, glass plate, electrodes, forceps, and scissors must be thoroughly cleaned to assure correct readings. Containers must be tightly covered to prevent cross contamination. Utmost care must be taken to prevent accidental contamination of the Pyranol, the paper sample or the equipment.

TEST SPECIMEN:

The test specimen shall consist of stacked layers of paper approximately 2 to 3 inches in width weighing 25 grams. In handling the specimens, great care should be taken that hands do not touch the paper to be tested.

PROCEDURE:

Clean the beaker, glass plate, electrodes, forceps and scissors in a trichlorethylene vapor degreaser and then wash with acetone solvent. Wash with a solution of Alconox (approximately 2%) or other suitable detergent in water until water will wet the entire surface uniformly. Rinse thoroughly with distilled water and dry at 105-125°C.

Note 1: Care should be taken to thoroughly clean all surfaces which will come in contact with the paper or Pyranol.

Note 2: The electrodes should be conditioned as specified in E4D2.

Stack sufficient sheets to make a weight of approximately 25 grams plus an outer wrap. Hold this pad with forceps, discard the top and bottom sheets and tie a cord around the stack to make a bundle. Weigh this bundle, cutting off pieces until a 25 ± 0.1 gram weight is obtained.

Note 3: Extreme care should be taken to protect the sample from any source of contamination including handling.

Dry the specimen at 150 ± 2°C for a minimum period of 16 hours.

Wrap a beaker with aluminum foil to shield it from light. Add 500 ml. of A1331C Pyranol and heat to 100 ± 2°C.

Note 4: Experience has shown that room temperature Pyranol will reach 100°C in approximately an hour when placed in an oven.

Note 5: The Pyranol used must have a minimum resistivity of 1000×10^9 ohm-cm and water content not exceeding 20 PPM before heating.

Place the beaker containing the hot Pyranol in the oven containing the dried sample. Grasping the sample by the string, hold it over the Pyranol and cut the string so that only the paper sample falls into the hot Pyranol. Discard the string.

Replace the aluminum foil cover and age the sample for 96 hours at 100 ± 2°C. Age a blank consisting of a similar beaker of the same Pyranol without a sample.

At the end of the aging period, place the resistivity electrodes in the beaker and measure the resistivity in accordance with E4D2.

CALCULATIONS:

Calculate the percent change in resistivity as follows:

$$\text{Percent Change} = \frac{-100\ (\text{Resistivity of Blank} - \text{Resistivity of Pyranol + Sample})}{\text{of Blank Resistivity}}$$

REPORT:

The report shall include the complete identification of the material tested and the change in resistivity.

R.A. Larrick
10/25/59

CLTX-20
Issued: 4-26-61
Revision: #1

THERMAL-CHEMICAL STABILITY TEST FOR IN-PROCESS CONTROL

I. SCOPE

This method describes a procedure for measuring the Thermal Stability of Pyranols, by exposing the Pyranol to heat in a sealed ampoule. The resulting chloride is determined potentiometrically and its magnitude is a measure of the Thermal Stability of the Pyranol.

II. APPARATUS AND EQUIPMENT

A. 32 x 200 mm Pyrex test tube
B. Glassworkers torch, glasses and various equipment
C. Oven operational to 250°C or over and controllable to ± 2°C
D. Balance accurate to 0.01 gram. Torsion Balance Style 1L9 adequate.
E. 50 ml. glass syringe, 6" 1/4 gauge stainless steel needle and stopcock.
F. Support for ampoules while in oven.
G. Beaker 200 ml. tall form. (Berzolius)
H. Magnetic stirrer and stirring bars (1" Teflon covered)
I. Microburette graduated in 0.01 ml. divisions
J. Silver electrode (billet type). Beckman #1261 adequate
K. Glass electrode. Beckman #4919V4 shielded adequate.
L. pH Meter suitable for use with glass electrode. - Model GS Beckman preferred because instrument has expanded scale for greatest sensitivity to incremental EMF changes. Somewhat less sensitive meter, such as, Beckman "Zeromatic" or Leeds Northrup line operated pH meter may be used.
M. Water bath - Use an individual glass water bath 150 mm in diameter 75 mm high (crystalizing dish) and containing 600 ml of water heated to 40°C ± 1°C.
N. Pipette 25 ml. Fisher #8-741 adequate.
O. Burette or pipette, graduated to deliver 0.5 ml.
P. Wash bottles for pure Acetone, Methanol, and water (Polyethelene)

Reagents

A. Methanol (chloride free) may be deionized through an ion exchange column or may be distilled from $AgNO_3$ crystals. Chloride ion concentration should be less than 0.01 ml of 0.005 N $AgNO_3$ per 100 ml. of Methanol.
B. Sodium Hydroxide, 0.1 N prepared by dissolving 4.0 grams of analytical reagent grade NaOH pellets in one liter of chloride free Methanol.
C. Sulfuric Acid, 50-50 by volume - Dilute analytical reagent grade concentrated Sulfuric Acid with chloride free (distilled or deionized) water.

CAUTION: Always pour acid into the water with constant stirring to prevent any dangerous build-up of heat.

D. Standard Silver Nitrate solution. 0.005N. Prepare by diluting ampoule of concentrated aqueous silver nitrate. Standardize against a pure chloride standard. (0.1N Silver Nitrate ampoules may be purchased from Anachemia, Montreal, Canada.)

E. Acetone, chloride free. Chloride ion concentration should be 0.01 ml of 0.005 N Silver Nitrate or less.
Acetone may be distilled from Silver Nitrate crystals if necessary. Solvents (Acetone & Methanol) should be checked for initial free Cl^- concentration to determine whether refining is necessary.

III. REFERENCES

Spec. E4C46-S1 - Unstable Chlorine Compounds in Askarels.

IV. PROCEDURE

A. Ampoule preparation

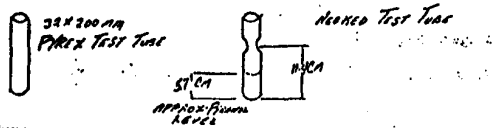
1. Thoroughly clean the 32 x 200 mm test tube with soap or detergent and water. Rinse thoroughly with deionized or distilled water to make sure all traces of cleaning agent are removed.

2. Place the cleaned test tube in an oven for at least three (3) hours at about 100°C.

3. After oven drying the test tubes are allowed to cool to comfortable handling temperature, 25-30°C.

4. By standard glass blowing procedures a glass rod is attached to the lip of the test tube and a neck formed at approximately 11.4 cm from the base. The neck should not reduce the diameter by more than half. See diagram below.

5. The test tubes may be prepared in advance, covered with aluminum foil and stored.



B. Test Procedure

1. Into a tarred-necked test tube weigh 50 grams ± 0.1 gram of Pyranol using a 50 ml. syringe with an 8" - 14 gauge needle for delivery. Care must be taken not to wet the neck of the test tube, because sealing temperatures will cause decomposition of the Pyranol.

2. The lower portion of the ampoule is covered with a wet paper towel to protect the Pyranol from the heat of sealing.

3. Seal the neck of the ampoule off with the torch by drawing. Check for leakage.

4. Do not extend the heating period beyond two (2) minutes, and do not apply the flame into the open test tube. Extreme temperatures and intense light can cause Pyranol breakdown, and high chloride results.

5. The hermetically sealed ampoule is placed in an oven operating at 240°C ± 2°C for two (2) hours.

6. When the two (2) hours have expired the ampoule is removed from the oven and allowed to come to room temperature.

7. After the ampoule has reached room temperature the neck is scored with a file and then the neck is struck sharply with a spatula to break off the end.

8. Empty the Pyranol into a clean 200 ml. Berzelius beaker.

9. Rinse the inside of the ampoule with 10 ml. of chloride free acetone, and empty this into the beaker.

10. With a 25 ml. pipette and automatic pipetter transfer 25 ml. of methanolic 0.1N sodium hydroxide to the beaker.

11. Add an additional 25 ml. of chloride free acetone

12. Place a clean Teflon covered stirring bar into the beaker. Do not let the hands touch the stirring bar. Use forceps or other transfer agent.

13. The beaker is now placed in a 40°C ± 1°C water bath. Sufficient water should be in the bath to cover the sample-occupied portion of the bath, i.e., about 4 cm. in a 7 x 15 cm. crystalizing dish.

14. The bath and sample are placed on a magnetic stirrer.

15. Stir the sample for five (5) minutes as fast as possible without causing pronounced splashing.

16. After five (5) minutes of stirring add 0.5 ml. of (50-50 by vol.) dilute sulfuric acid.

17. With a graduate add an additional 65 ml. of chloride free acetone.

18. Titrate for chlorides potentiometrically using the silver-glass electrode pair as per General Electric Test Method E4C46-S1 "Unstable Chlorine Compounds in Askarels".

19. Determine the reagent blank by performing the titration steps above, but omitting the Pyranol. i.e., Add 35 ml. of acetone and 25 ml. of methanolic 0.1N NaOH to a 200 ml. Berzelius beaker, stir in a 40°C bath for 5 min., acidify with 0.5 ml. of (50-50) dilute sulfuric acid, add an additional 65 ml. of Acetone, and Titrate for Cl^-.

Determine the Pyranol blank by weighing 50 grams of the original Pyranol into a 200 ml. Berzelius beaker, adding 35 ml. of chloride free Acetone plus 25 ml. of methanolic 0.1N sodium hydroxide and repeating steps #12 through #18. This represents in ppm of Cl^- the chemical contribution to [illegible] Determine Blank & Sub.

20. If the Pyranol blank falls within a 2 sigma (σ) deviation from the average ($\bar{x}$) value use the average as the Pyranol blank.

21. If the value falls outside the 2 sigma (σ) limits use the actual value as the Pyranol blank.

Calculations

$$\text{Gross PPM Chlorides} = \frac{\text{net ml. } AgNO_3 \times \text{normality of } AgNO_3 \times 35460}{\text{Weight of Sample in grams}}$$

$$\text{Pyranol Blank PPM} = \left(\frac{\text{net ml. } AgNO_3 \times \text{normality of } AgNO_3 \times 35460}{\text{Weight of Sample in grams}}\right) - \text{Reagent Blank}$$

22. Record the net thermal chloride value and the Pyranol blank in ppm Cl^-
Net thermal Cl^- = Gross Thermal - Chemical Cl^- - (Pyranol blank + Reagent Blank)

23. If the net Thermal Chloride value deviates from the 2 sigma (σ) limit a second determination should be made. If the second value falls outside the control limits (2σ) the Pyranol will not be released without the supervisors approval.

24. New (2σ) two sigma and ($\bar{x}$) average values for the net Thermal Chlorides and Pyranol blank (chemical Cl^- contribution) will be calculated every thirty days or every fifty-two (52) samples, whichever is most frequent.

25. The new control limits and any obvious trends in results obtained will be brought to the attention of the unit supervisor.

J. F. Tracy

CLTM-37
Date Issued: 4-5-61
Revised:

VOLUME RESISTIVITY OF DIELECTRIC LIQUIDS

Principle:

The volume resistivity of a dielectric liquid is an expression of its ohmic resistance times a constant which is representative of the volume occupied.

Object:

The D.C. resistivity is a rough gauge, easily measured, of material purity. In order to obtain a continuity between readings like conditions must be met with each consecutive set of readings, i.e., temperature, cell condition, exposure to light, etc., must be similar in each instance. The values obtained are determined on the basis of an arbitrary set of conditions previously agreed upon. Altering any of these conditions lessens the continuity between readings.

Reference:

G.E. E4D2-S2
"Physics" E. Hausman and E. P. Slack
D. Van Nostrand Co., Inc. New York, New York (1948)

Equipment:

(1) G. E. cylindrical resistivity cells, Catalog # 1,559,663, nickel plated brass.

(2) Laboratory thermometer operable to 0 - 100°C or 0 - 200°C ± 1°C and meeting ASTM Spec. E1.

(3) "Pyrex" glass spacer plate for above resistivity cells.

(4) 800 ml "Pyrex" beaker.

(5) Heating mantle with auto transformer for voltage adjustment or hot plate. The heating mantle is preferable and should closely conform to 800 ml size.

(6) Resistance meter:
(a) Electronic Instruments, Ltd. 20 million megohmeter.
(b) General Radio 544B.

Procedure:

Before testing may begin the resistivity cells, glass spacer, beaker and thermometer should be thoroughly cleaned. The following procedure should be

used in order to obtain a continuity of results between labs. If other cleaning methods are used, there may not be a continuity of readings between labs or with past results.

1. All parts such as the beaker, spacer, cells, etc., should be vapor degreased prior to washing.

2. The glass spacer, beaker and thermometer may be washed manually with "Alconox" or may be washed by soaking in Oakite bottle wash solution.

3. They should be rinsed with tap water and distilled water.

4. Each item should be checked for wettability with distilled water. Complete wetting should take place with no soil or slick spots remaining.

5. The resistivity cells should be washed by hand with "Alconox" or other non-abrasive cleaner. A soft cloth or paper toweling may be used to facilitate cleaning. Do not scratch or mar the bright nickel surface. Badly oxidized or scratched cells with plating worn thin should be replaced. Hand washing is the only accepted way to clean these cells. Do not put in bottle soak or other strong caustic.

6. After washing, the various parts are put into 100°C oven to dry for one to two hours. They should not be left in the oven overnight as this will form an oxide and require rewashing.

7. After washing, the cells are assembled in the beaker and the air capacitance determined as per CLTM-35.

8. The cell constant (K) is obtained by multiplying the capacitance in uuf x 11.3.

$$K = C\ (uuf)\ 11.3$$

9. Prior to obtaining readings on samples the cells should be conditioned in the material they are destined to be used in. Normally three or four rinsings and electrifications at 100°C will bring the cells to a steady state. If the resistivity meter is calibrated and steady state readings cannot be obtained, the cells may have to be recleaned. A set of cells should be reserved for one dielectric liquid and are not interchangeable between dielectric liquids.

10. Prior to placing liquid to be tested into the hot cells (100°C), the liquid should be heated to 100°C in an oven or heating mantle. This will bring cells and dielectric to the same temperature range and eliminate fluctuations in resistivity brought about by temperature cycling.

11. Fill the cell with the heated liquid to be measured.

12. Bring to temperature specified - usually 100°C ± 1°C.

13. Attach leads (a) shielded lead to inner electrode (b) guard wire to external guard, i.e., foil or insulated metal beaker (c) third lead to outer electrode.

14. Make sure resistivity meter is calibrated or zeroed prior to use.

15. Charge sample for 30" (a) on Electronic Instrument depress "A" button (b) on G.R. bridge switch selector knob to charge.

16. Bring meter into the circuit (a) on Electronic Instrument depress "B" button while leaving "A" depressed (b) on G.R. bridge switch to operate.

17. After a total of one minute electrification obtain reading and calculate resistivity.

P (resistivity) = 11.3 x C x R
P = KR where
K = cell constant
R = resistance in megohms
11.3 = conversion factor from electrostatic units.

18. Report results as follows:
a. Calculated resistivity usually reported at x 10^{9} ohm cm for Pyranols; x 10^{12} ohm cm for oils.
b. Record temperature at which measurement was taken. Most dielectrics are measured at 100°C.
c. Record voltage.
d. Identify material under test, i.e., 1499, A13B1C or AR #.

19. Cells may be stored in the dielectric liquid until the next use. They should be covered and shielded from exposure to ultra-violet light.

Deviation of Cell Constants:

In arriving at a constant for resistivity cells the following should be taken into consideration:

Cylindrical Cells:

The resistance between two coaxial cylinders of length l. and radii r_1 and r_2 is:

1. $R = \dfrac{\ell_n \dfrac{r_2}{r_1}}{2\pi l} \rho$

ρ = resistivity
ℓ_n = natural log
$2\pi l$ = area
R = resistance (ohms)
K = cell constant

2. or $K = \dfrac{2.303 \log_{10} \dfrac{r_2}{r_1}}{2\pi l}$

or the resistivity ρ may be written as the product of a cell constant K and the resistance R.

3. $\rho = KR$ where $K = \dfrac{2\pi l}{2.303 \log_{10} \dfrac{r_2}{r_1}}$ cm.

The capacitance between these same two cylinders is:

4. $C = \dfrac{1}{4.15 \log_{10} \dfrac{r_2}{r_1}}$ = uuf

If we divide C eq 4 by K eq 2, we obtain

$K = \dfrac{8.30\pi}{2.303}$ C or $3.6\pi C = 11.3 \times C$

K = cell constant
C = cell capacitance in uuf.

The determination of cell constant for Parallel Plate capacitors:

$C = \dfrac{EA}{4\pi S}$

C = Capacitance esw
E = Permitivity = 1
A = Area
S = Thickness of dielectric or space between plates

To convert C in esu to m.f., the divisor is multiplied by 9×10^5. Then, in order to convert the product to a familiar form commonly used uuf, the whole is multiplied by 10^6.

$C = \dfrac{EA \times 10^6}{4\pi S \times 9 \times 10^5}$ or $C = \dfrac{EA \times 10}{4\pi S \times 9} = \dfrac{EA}{S} \times \dfrac{1}{3.6\pi}$

$3.6\pi = \dfrac{EA}{SC}$ $\qquad 11.3 = \dfrac{EA}{SC}$

$C \times 11.3 = \dfrac{EA}{S} = \dfrac{EA}{S} = K$ (cell constant)

J. F. Tracy

MONS 034882

Memo #1

INFRARED EXAMINATION
of
DIELECTRIC LIQUIDS

Apparatus:

Spectrometer, Perkin-Elmer, Model 21 or other double-beam instrument, preferably with Ordinate Scale Expansion

Following NaCl cells:

1 matched pair of sealed cells (0.1mm)

1 0.025 mm sealed

1 demountable cell (capillary film)

Charts, full scale (5 cm/u) and Keysort card (1cm/u)

Test Specimen:

The test specimen shall consist of an aliquot (usually 1 qt.) representative of the material.

Procedure:

Adjust spectrometer to the following conditions:

Resolution - 927

Response - 3 sec. full scale

Gain - 5-1/2(nominal)

Speed - 1 min/micron (u)

Suppression - 8

Source current - 0.3 amp.

Place undiluted samples in sealed 0.1 mm, 0.025 mm and capillary film cells. Using full scale chart and 0.1 mm cell record 2 - 15 micron region. Use 0.025 mm and capillary films to resolve totally absorbing bands observed. A NaCl crystal of suitable thickness is used as reference for these runs.

On the same chart record a differential spectrum with 0.1 cm matched cells, using the appropriate, standard in the reference beam. Repeat the differential analysis at 1 cm/micron on a Keyport card.

Examine the differential spectrum and note any marked departures from previous differential analysis. An aliphatic band at 3.4-3.5 microns and diphenyl bands at 13.5 and 14.5 microns should be absent for all Pyranol samples. In addition, 1499 Pyranol in 1476 Pyranol can be detected at 9.95 -10 microns, and 1476 Pyranol in 1499 Pyranol by a band at 7.45 - 7.52 microns.

Report:

The report shall include the complete identification of the material tested, the infrared chart number and any abnormalities and/or changes evident in the material.

Treated Foil Test for Quality of Dielectric Paper

Purpose:

The purpose of this procedure is to obtain data on wound and treated units prior to accepting paper as an indication of paper quality. This technique can also be used in evaluating new machines and processes, and new types and grades of paper. The tests normally performed on the units are dry absorption, treated power factor at two temperatures and D.C. dielectric strength.

Procedure:

Thirteen units are wound according to the marked thickness and Table I below. The information below is submitted with the wound units.

Sample number
Vendor
Lot number
Specifications
Thickness
Foil Vendor
Foil lot number
Turns
Feet
Winding date
Winding operator
Winding machine
Roll number

The winder assigns sample numbers consecutively with an "H" prefix, fills in the card and delivers the wound units, unused paper and record cards to the assembler.

The assembler assembles the rolls to covers (1175:15G1AC251) and cans (1CM1C01). Three of each group in the box with the excess paper, after marking the "H" number on the cover are retained. The ten remaining units are seamed, tagged, and sent to treat.

After the treat the ten units go to Q.C. Five are hand labeled, tags removed, and placed in a 125°C oven [illegible] overnight. The next morning the power factor and capacity are read at 300 volts-per-mil. The units are cooled overnight and read again the next day at 25°C.

When the data is available certain calculations are made. The power factors for the two temperatures and the breakdown results are averaged. From the 125°PF two quantities will be subtracted. The first, called a polar component, is determined from the type of pulp and density according to the following:

KMB pulp (A50PC14) P= .230 x density

A graph of this one equation is made available so that calculations are not necessary. The second quantity called the ionic component is a function of the absorption reading as follows:

I = .064 x Absorption

The 125°PF is obtained by subtracting a foil loss which is a function of foil length and capacitance (the winder records foil length from an unwound unit) according to the following:

t = 25°C	D = LC/4446	L = length in feet
65	D = LC/3750	C = Capacity in uf
100	D = LC/3310	D = % D foil loss
115	D = LC/3151	
125	D = LC/3052	

From this difference the above two components are deducted and the residual is termed "excess power factor". The calculation of the polar component is adjusted so that the excess is very close to zero for a normal lot. The natural variation in power factor and absorption sometimes cause the calculated excess to be negative. A hand tally is maintained of the averaged and calculated results of each lot.

The paper lab maintains charts by density of four of the results: 125° PF, 25% PF excess power factor and breakdown. Periodically distributions are drawn up and 95% (2σ) limits are determined for control limits.

Calibration:

The oven used to test for power factor should have a recorder-controller. Four units are read. A standard of some type is needed for the absorption system that will detect inaccuracies when the system is operated, rather than separate checks on each of the components. For power factor lead losses, thirteen (13) units were selected with approximately the same power factor levels but with a full range of capacitance. If the difference between the actual power factor reading and a standard value correlates with capacitance, a lead loss is indicated.

TABLE I

FACTORY TRIAL WINDING INSTRUCTIONS

Mandrel - .375"
Foil - (2) .25M x 1.5"
Paper - (4) 1.75" x thickness as required
Taps - 9811913 - P4
Turns - As follows:

Thickness	Foil Turns*	Tap Turns**	Feet***
.20M"	450	288	113.
.25	390	250	98.
.30	344	220	86.3
.35	308	198	77.3
.40	278	178	70.0
.45	254	163	63.8
.50	234	150	58.8
.55	216	138	54.3
.60	202	130	50.7
.66	188	121	46.8
.75	167	107	42.0
.80	158	101	39.8
.88	145	93	36.5
1.00	130	83	32.7

*Foil turns = $\frac{250W}{t_f + 2t_p}$

**Tap turns = $\frac{\text{Foil turns}}{1.56}$

***Feet = $\frac{73.4}{t_f + 2t_p}$

W = Can width = 1.17"
t_f = Foil thickness = .25M"
t_p = Paper thickness = variable

MONS 034888

EPOXIDE 206
VINYLCYCLOHEXENE DIOXIDE

1. SPECIFIC GRAVITY
   a) Determine at 20°C by means of a hydrometer calibrated to give the apparent specific gravity at 20/20°C and capable of being read to the nearest 0.0005 unit.
   b) Maintain the constant temperature bath at 20 ± 0.05°C.
   c) Reference 5-B2-4

2. VISCOSITY
   a) Determine the kinematic viscosity at the specified temperature by means of a calibrated capillary-type viscometer having the required centistoke range.
   b) Use either an Ubbelohde or an Ostwald-Fenske viscometer.
   c) Report the viscosity in centistokes.

3. TOTAL EPOXIDE
   CAUTION: This procedure involves the use of perchloric acid. Do not use this reagent unless aware of its potential hazards.
   a) Hydrogen bromide solution, 0.5 N in acetic acid By means of a graduate, carefully add 67 ml of c.p. bromine to two liters of glacial acetic acid. Add reagent grade phenol in 10-gm increments until the solution becomes light straw in color (approximately 100 gm) and add 10 gm in excess. Mix the solution after each addition of the phenol and allow the final solution to stand overnigh before using.
   b) 0.2 N Sodium Acetate in acetic acid Dissolve 16 gm of anhydrous sodium acetate in sufficient glacial acetic acid to make one liter of solution. Standardize this solution against standard 0.2 N perchloric acid using 1.0 per cent crystal violet indicator in acetic acid.
   c) Procedure Prepare a sufficient number of clean dry 250-ml iodine flasks to perform all sample and blank determinations in duplicate.
   d) Carefully pipet 25 ml of the hydrogen bromide solution into each of the flasks, using the same pipet for each transfer.
   e) Reserve two of the flasks for the blank determination.
   f) Into each of the other flasks introduce 0.3 to 0.4 gm of the sample weighed to the nearest 0.1 mg by means of a suitable weighing pipet and swirl to effect solution.

g) Stopper the flasks, using 5 ml of glacial acetic acid as the liquid seal, and allow the samples to stand with the blanks at room temperature for 15 minutes.
h) Carefully remove the stopper from each flask and wash down the stopper and inside walls of the flasks with 25 ml of glacial acetic acid.
i) To each flask add 5 or 6 drops of crystal violet indicator and titrate with standard 0.2 N sodium acetate in acetic acid to the first bluish-green end point.
j) Calculation

$$\frac{(B - A)N \times 1.6}{\text{gm sample}} = \text{total epoxide, \% by weight, as oxirane oxygen}$$

A = ml of N normal sodium acetate required for the sample

B = average ml of N normal sodium acetate required for the blank

4 ACIDITY
a) Introduce 50 gm of the sample weighed to the nearest 0.1 gm into a 250-ml Erlenmeyer flask containing 50 ml of methanol which has been previously neutralized to a faint pink end point using 1.0 per cent phenolphthalein indicator in methanol. Swirl to effect solution.
b) Add a few additional drops of the indicator and titrate with standard 0.1 N alcoholic potassium hydroxide to the first pink end point permanent for at least 15 seconds.
c) Calculation

$$\frac{AN \times 6.0}{\text{gm sample}} = \text{acidity, \% by weight as acetic acid}$$

A = ml of N normal KOH required

5 TOTAL UNSATURATION
a) Wijs solution Dissolve 13 gm of resublimed iodine in 1000 ml of glacial acetic acid. Gentle heat may be necessary to effect solution. Allow to cool and remove approximately 200 ml of the solution. Pass dry chlorine gas into the remainder of the iodine solution until the original titration is not quite doubled. Perform the titration as directed below. A characteristic color change takes place in the Wijs solution when the desired amount of chlorine has been added. This may be used as an aid in judging the end point. Add a small excess of chlorine and bring back to the desired titration by addition of some of the original iodine solution. Into respective 500-ml Erlenmeyer flasks pipet 25 ml of the solution before addition of the chlorine and 25 ml of

the solution after addition of chlorine. Add 20 ml of 15 per cent potassium iodide solution to distilled water and 100 ml of distilled water to each flask. Titrate the contents of each flask with standard 0.1 N thiosulfate until the yellow color almost disappears. Add two ml of 1.0 per cent starch indicator solution and continue titrating until the blue color disappears.

b) Procedure Heat all samples which are solid or semi-solid or contain turbidity under an infrared heat lamp or in a hot water bath until completely homogeneous, and mix well.

c) Prepare a sufficient number of clean dry 500-ml glass-stoppered Erlenmeyer flasks to perform all blank and sample determinations in duplicate.

d) To each flask add 20 ml of reagent grade carbon tetrachloride by means of a graduate.

e) Reserve two of the flasks for the blank determination.

f) Into each of the other flasks introduce 14 to 16 gm of the sample weighed to the nearest 0.01 gm and swirl to effect complete solution.

g) Into each flask pipet 25 ml of the Wijs solution using pressure rather than suction to fill the pipet and again swirl to effect solution.

h) Allow the flasks to stand in the dark for 30 minutes at room temperature.

i) To each flask add 20 ml of the 15 per cent potassium iodide solution, and 100 ml of distilled water.

j) Titrate immediately with standard 0.1 N sodium thiosulfate until the yellow color almost disappears. Add two ml of the starch indicator solution and continue titrating to the disappearance of the blue color. If the sample titration is less than 50 per cent of the blank repeat the determination using a smaller size sample.

k) Calculation

$$\frac{(B - A)N \times 12.69}{\text{gm sample}} = \text{total unsaturation as iodine number}$$

A = ml of N normal $Na_2S_2O_3$ required for the sample

B = average ml of N normal $Na_2S_2O_3$ required for the blank

6. COLOR

a) Transfer 100 ml of the sample to one of two matched tall-form Nessler tubes.

b) Fill the second tube to the mark with the platinum-cobalt standard that appears to match the color of the sample.

c) Compare the colors of the sample and the standard by viewing vertically down through the tubes against a white background.

d) Determine the exact color of the sample by successive replacement of the standard in the second tube until an exact match is obtained.
e) Reference : 5-J1-1

SUSPENDED MATTER
a) Invert a bottle of the sample and examine by transmitted light.

DEVELOPMENT DEPARTMENT
TECHNICAL CENTER
South Charleston, West Virginia