MONSANTO CHEMICAL COMPANY

ANNISTON, ALABAMA

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Anniston

Comparison of Anniston and General Electric Methods for Free Chlorides in Aroclors

Because of St. Louis' difficulty with free chlorides and Anniston's recent trouble in maintaining our traditional low free chloride content in liquid Aroclors, considerable attention has been focussed on the method of determining free chlorides in this material. On several occasions our analysis has been lower than that of General Electric, and one lot (#1486) was actually rejected by G. E. because of high chlorides whereas our analysis was within specifications. Several weeks ago Mr. Loeven discussed these facts and requested an exhaustive study of our analytical control, directed toward obtaining a comparison of our method with that of General Electric, evaluating both, and if possible developing an improved method for determining free chlorides in liquid Aroclors. Herein are reported findings and conclusions on all points but the last, which is still under investigation.

Methods of Analysis

1. General Electric - Method described in G. E. Folder #DF-65206 dated 8/30/45, modified according to supplementary methods attached to communication of F. H. Wheelock to H. Weaver 9/3/47, further modified by memo of R. L. Jenkins to R. Loeven 11/14/47. Basically the method is as follows:

A. All apparatus is washed and rinsed until chloride-free

B. 50 g. Pyranol is extracted with 75 ml chloride-free water, allowed to settle

C. 10 ml extract is shaken with an equal volume of ether in a test tube until the water layer shows no effect under the Tyndall Beam test

D. 3-5 drops acidified $AgNO_3$ added without stirring to sample and standards, and the beam from a small flashlight ("Penlite") is passed through the side of the tubes, observed through the sides at right angle to the beam. This observation is made after 15 sec. and before 45 sec. after addition of the $AgNO_3$.

E. Chloride content of sample extract must produce no more beam effect than a standard containing 0.10 ppm. NaCl. "This amount is the minimum that can be detected."

2. Anniston Method

A. 200 g Aroclor is extracted with 50 ml distilled water. The laboratory is accustomed to using distilled water which is not completely chloride-free, though this added chloride tends to cancel out since both samples and standards are diluted with the same water.

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2. Anniston Method continued:

B. The water extract is decanted and clarified by shaking with an equal volume of ether. A 10 ml aliquate is transferred to a test tube.

C. Standards are prepared equivalent to 0.050, 0.075, 0.10, 0.15 ppm. Chlorides (based on size sample and aliquate taken). These are placed in 10 ml test tubes of same type as used for the sample.

D. To each test tube is added 0.5 ml acidified silver nitrate, and the turbidity of sample is compared with standards by viewing vertically through the depth of the solution against a black background.

Investigational Procedure

1. A survey was made of recent lots, comparing Anniston and G. E. reports.
2. A still was set up to obtain chloride-free water.
3. Regular lots of liquid Aroclors were analyzed by each of three methods: General Electric, Anniston, Anniston using chloride-free water.
4. The General Electric method in each case was extended to five minutes to determine effect of time on apparent chloride content.

Evaluation of Results

1. Apparently Anniston had no difficulty making low-chloride Aroclor before lot #1452, corresponding to June 7, 1947. Since no change was made in analytical procedure, a search must be made elsewhere to discover the cause of the abrupt rise in free chlorides after this date.
2. It is interesting to note that in the original copy of G. E. methods received by this laboratory, the free chloride test, carrying date 5/29/40, requires comparison against a 0.2 ppm. standard (because of slightly different concentration of extract) and states that "This amount of chlorides is the minimum amount that can be visually detected." But the method mentioned above, dated 8/30/45 calls for a standard solution containing 0.10 ppm. chloride and contradicts the declaration of the previous official procedure by stating "This amount is the minimum that can be detected". Obviously both pronouncements cannot be true. It is our observation that a Tyndal Beam effect can be observed in a 0.10 ppm. solution, but that its use requires considerable dexterity and practice from the operator.
3. The Tyndall Beam effect of the General Electric method changes with time and this variation is neither consistent nor predictable.

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Evaluation of Results Continued:

4. Without much practice it is difficult to compare the sample with several standards within the very narrow time limits prescribed by the Tyndall Beam test, yet the operator must adhere closely to these limits to avoid errors caused by the time sensitivity of the test. This comparison is further complicated by the fact that 0.10 ppm. gives such a faint Tyndall Beam effect that it can be easily distorted by fingerprints on the tube, air bubbles or suspended dust or lent fibers in the liquid.
5. The Anniston method with redistilled water checked the General Electric method (interpolated below 0.10 from the curves) in all cases but lot #1494 and #3 tank, in which runs the Tyndall Beam gave higher chlorides.
6. In 75% of the cases (6 lots) the regular Anniston method failed to check the General Electric methods and in 63% (5 lots) failed to check the Anniston method using redistilled water. Apparently Anniston has followed this practice without challenge only because G. E. reports nothing less than 0.10 ppm.

Conclusions

1. The General Electric method is adequate as a maximum specification test, and G. E. uses it only as such - to report chlorides as less than, equal to, or greater than 0.10 ppm.
2. The Anniston method as practiced using only single-distilled water must be discontinued.
3. The Anniston method using chloride-free water checks the G. E. method satisfactorily.
4. Considering the delicacy and limited usefulness of the Tyndall Beam method, the modified Anniston method is to be preferred as a general technique. If the Tyndall Beam is used it should be only for the same purpose and reported in the same way as by General Electric.
5. If this determination can be made instrumentally, thus eliminating errors in judgement, it will be considerably improved. This possibility is being studied.

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Comparison of Anniston and General Electric Reports of Free Chloride

Lot No.	Anniston	General Electric
1473	.075	Less than 0.10
1472	.10	" " .10
1470	.075	" " .10
1469	.10	" " .10
1466	.05	" " .10
1454	.10	" " .10
1450	.05	.10
1448	.15	Less than .10
1452	.075	" " .10
1445	.050	" " .10
1450	.050	" " .10
1441	.050	" " .10
1440	.050	" " .10
1430	.075	" " .10
1427	.050	" " .10
1436	.050	" " .10
1426	.050	" " .10
1422	.050	" " .10
1420	.050	" " .10
1419	.025	" " .10
1404	.050	" " .10
1406	.050	" " .10

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Comparative Free Chloride Content by Various Analytical Methods

Lot#	Anniston Regular	Anniston Distilled	General Electric Method Recorded as by (1) G. E.	General Electric Method Interpolated (2) from curves
1248 (Aroclor 1260)	.075	.075	<.10	.075
1249	.10	<.05	<.10	.050
#3 Tank	.05	.05	.10	.10
1492 (Aroclor 1254)	.10-	<.05	<.10	.05
1493	.10-	<.05	<.10	.04
1494	.10+	<.05	<.10	.075
1496	.05	.05	<.10	.05
#1 Tank	.10-	.05	<.10	.05

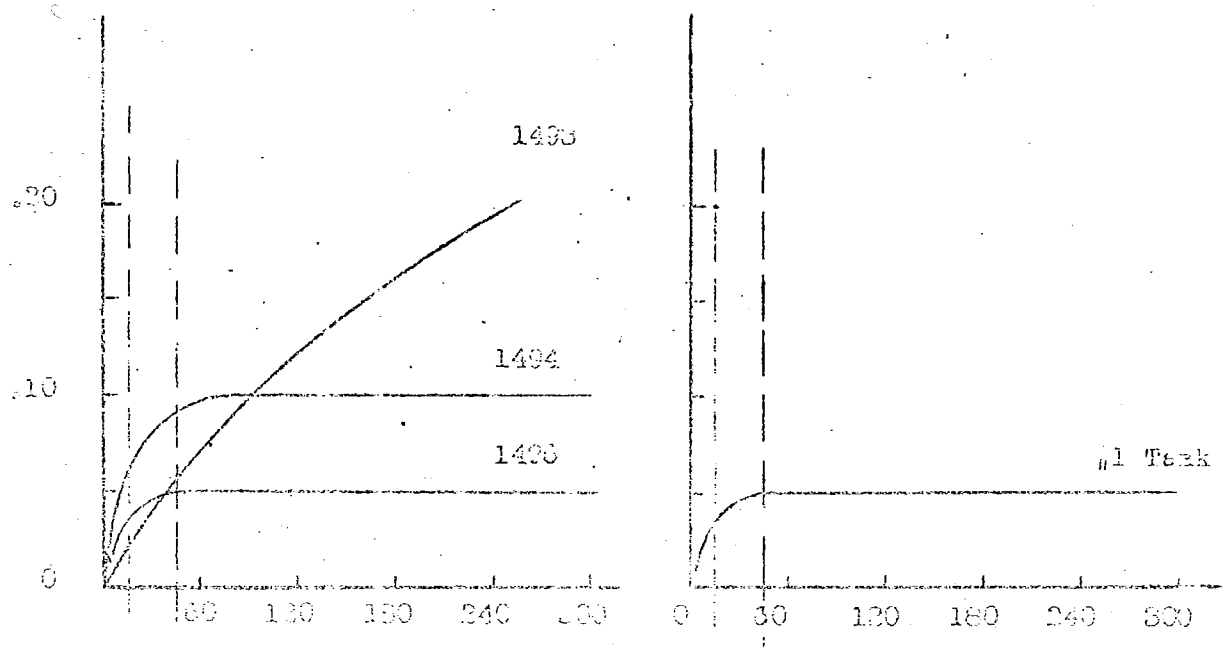
Notes: (1) General Electric reports values below 0.10 only as "less than 0.10".
(2) Taken from curves at 30 sec., midpoint of G. E. time limits

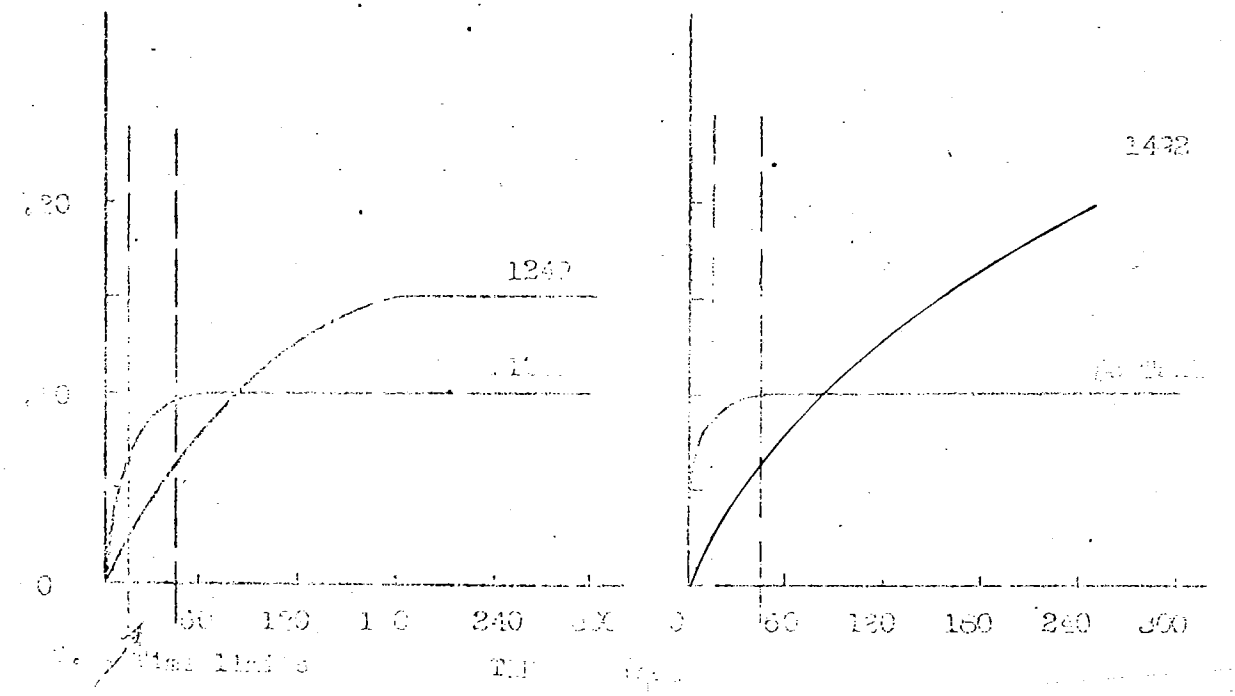
Variation of Apparent Free Chloride with Time General Electric Method

Lot#	Time (sec)								
	15	30	45	60	90	120	180	240	300
1248	<.10	<.10	<.10	<.10	.10	.10	.10	.10	.10
1249	<.10	<.10	<.10	.10	.1.10	.10-.15	.15	.15	.15
#3 Tank	<.10	.10	.10	.10	.10	.10	.10	.10	.10
1492	<.10	<.10	<.10	.10	.10	.15-.20	.15-.20	.20	
1493	<.10	<.10	<.10	<.10	.10	.10-.15	.15	.20	
1494	<.10	<.10	<.10	<.10	.10	.10	.10	.10	.10
#1 Tank	<.10	<.10	<.10	<.10	<.10	<.10	<.10	<.10	<.10

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