

INTER-OFFICE MEMO **TENNECO CHEMICALS, INC.**

TO M. Freifeld

AT Flemington

DATE April 3, 1973

FROM H.B. Carr

AT Burlington

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SUBJECT Lead Assay

*See y/b
LWR*

As we concluded at an All-Disc task force meeting on 11/17/72, the atomic absorption assay method for lead is inadequate due to erratic results. Since that time, we have been successful in developing a gravimetric assay technique that is accurate and reproducible for lead analyses of dibasic lead stearate, tribasic lead sulfate and compound dry blends containing these stabilizers.

The method is based on the digestion of a sample in 1:1 $\text{HNO}_3:\text{H}_2\text{SO}_4$, followed by oxidation with H_2O_2 , driving off the HNO_3 and precipitating the lead as the sulfate. The lead sulfate is filtered through a tared gooch crucible and dried to constant weight from which the lead content is calculated (factor is 0.683). This method will be adopted as a standard procedure.

In the attached Table I (Lead Analyses), assay results are shown for dibasic lead stearate, tribasic lead sulfate and compound dry blend. It will be noted that all dibasic lead stearate samples are in-specification but that the Eagle-Picher tribasic lead sulfate samples are somewhat low. In a round robin interchange, Eagle-Picher confirmed the low lead assays and has stated that appropriate corrective action will be taken. The compound dry blend results indicate a fairly wide spread in results (0.61% - 0.82%) versus a theoretical result of 0.74%. A laboratory-prepared dry blend assayed at 0.73%. The dry blend assay results are limited and more data is required to assess actual plant control.

The lead assay method is currently being established in Burlington. Each raw material lot will be assayed for conformance to specification. Additional dry blend analyses will be performed on each 3805D lot produced, the target being six analyses per lot.

H.B. Carr

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Attachment.

HBC/jst

TABLE I
LEAD ANALYSES

Dibasic Lead Stearate

NL Industries

DS 207

Specification for Pb: 50.4% - 52.2%

<u>Compound Lot Number</u>	<u>Sample</u>	<u>Result</u>	<u>DBLS Lot Number</u>
Lot 7-112849	Mix 1	50.8% Rerun (51.1%)	NL Lot 2850
"	Mix 23	52.2%	" " "
"	Mix 49	51.3%	" " "
"	Mix 59	51.3% Rerun (51.2%)	" " "
"	Mix 137	52.1%	" " "
Lot 7-013000	-	51.8%	NL Lot 2875
Lot 7-023102	-	51.4%	NL Lot 2879

Tribasic Lead Sulfate

Eagle-Picher

Epistatic 100

Specification for Pb: 83.5% - 84.5%

NL Industries

Tribase

Specification for Pb: 82.2% - 83.3%

<u>Compound Lot Number</u>	<u>Sample</u>	<u>(Tenneco) Result</u>	<u>(Eagle-Picher) Result</u>
Lot 7-112849	Mix 1	81.3% (EP Lot 27A-505)	81.9%
"	Mix 23	81.2% Rerun (81.5%) (EP Lot 27A-505)	82.0%
"	Mix 49	81.0% (EP Lot 27A-505)	81.8%
"	Mix 60	82.7% (NL Lot 172255A)	-

TABLE I
LEAD ANALYSES
(continued)

Dry Blend

Pb content based on formulation: 0.74%
(0.8% DBLS; 0.4% TBLS)

<u>Compound Lot Number</u>	<u>Sample</u>	<u>Result</u>
Lot 7-112849	Mix 7	0.82% Rerun (0.85%)
"	Mix 8	0.70%
"	Mix 9	0.75%
"	Mix 10	0.85%
"	Mix 17	0.61% Rerun (0.64%)
"	Mix 64	0.73%
Lab-Prepared Dry Blend		0.73%
TBLS 82.7%		
DBLS 51.3%		