MONSANTO CHEMICAL COMPANY
W. G. KRUMMRICH PLANT LABORATORY

STANDARD ANALYTICAL METHOD

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11-19-70	1 OF 2	P-2286
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ATTACHMENTS: Sample Chromatogram		

PRODUCT: Pyranol/Therminol (returned)

TEST: Low Boilers by GLC

THIS FORM [ ] SUPERSEDES METHOD _____ ISSUED ON NEW
[ ] REVISES INSTRUCTIONS

SCOPE

This procedure determines low boilers in returned pyranol or therminol. The area of the low boilers are normalized. The rest of the GLC curve is compared to a standard curve to spot irregularities in contaminated material.

EQUIPMENT

1. GLC, Varian-Aerograph Model 202-1C, dual-column linear temperature programmed, equipped with a 1 MV, 1 second response time (or better) recorder and a 120 rpm Disc, integrator (specify when ordering). Varian-Aerograph, 205 W. Touhy Ave., Park Ridge, Ill. 60068, (or equivalent).

2. GLC columns, two 4-foot by 1/4 inch O.D. stainless steel, Metal Goods Corp., St. Louis, Missouri (or equivalent).

3. Column Packing, 3% GE-SE30 on Chromosorb G., A/W, DMCS treated, 60/80 mesh, Analabs, Inc., P. O. Drawer 5397, Hamden, Conn., 06518 (or equivalent).

4. Helium supply and regulator to deliver 50# pressure at 100 cc/minute.

5. Syringe, Hamilton, No. 701N with 2 inch needles. Fisher Scientific Co., 1241 Ambassador Blvd., St. Louis, Mo., 63132

REAGENTS

1. Benzene, Reagent Grade, Cat. No. B-245, Fisher Scientific Co.

SAFETY

Benzene is flammable. Keep away from open flames and spark sources.

Pyranol/Pydraul/Therminol may contain toxic contaminants when returned. Handle with care and wash contacts with soap and hot water immediately.

INSTRUMENT CONDITIONS

Injection Port	330 ± 5°C
Detector Oven	345°C

Bridge Current	160 ma
Flow Rate	100 cc Helium/min.
Column Oven (initial)	80°C
Temperature Program	10°/min. to 340°C

DETERMINATION

1. Dilute 5 ml of sample with an equal volume of benzene. Stopper and shake to mix well.

2. Bring the instrument to the conditions specified and inject 5 ul of sample/solvent mix.

3. Start the temperature program immediately and turn on the integration device. An attenuation of 8:1 should keep all components on scale (except the benzene).

4. After all components have eluted determine the area under all the peaks (excluding benzene).

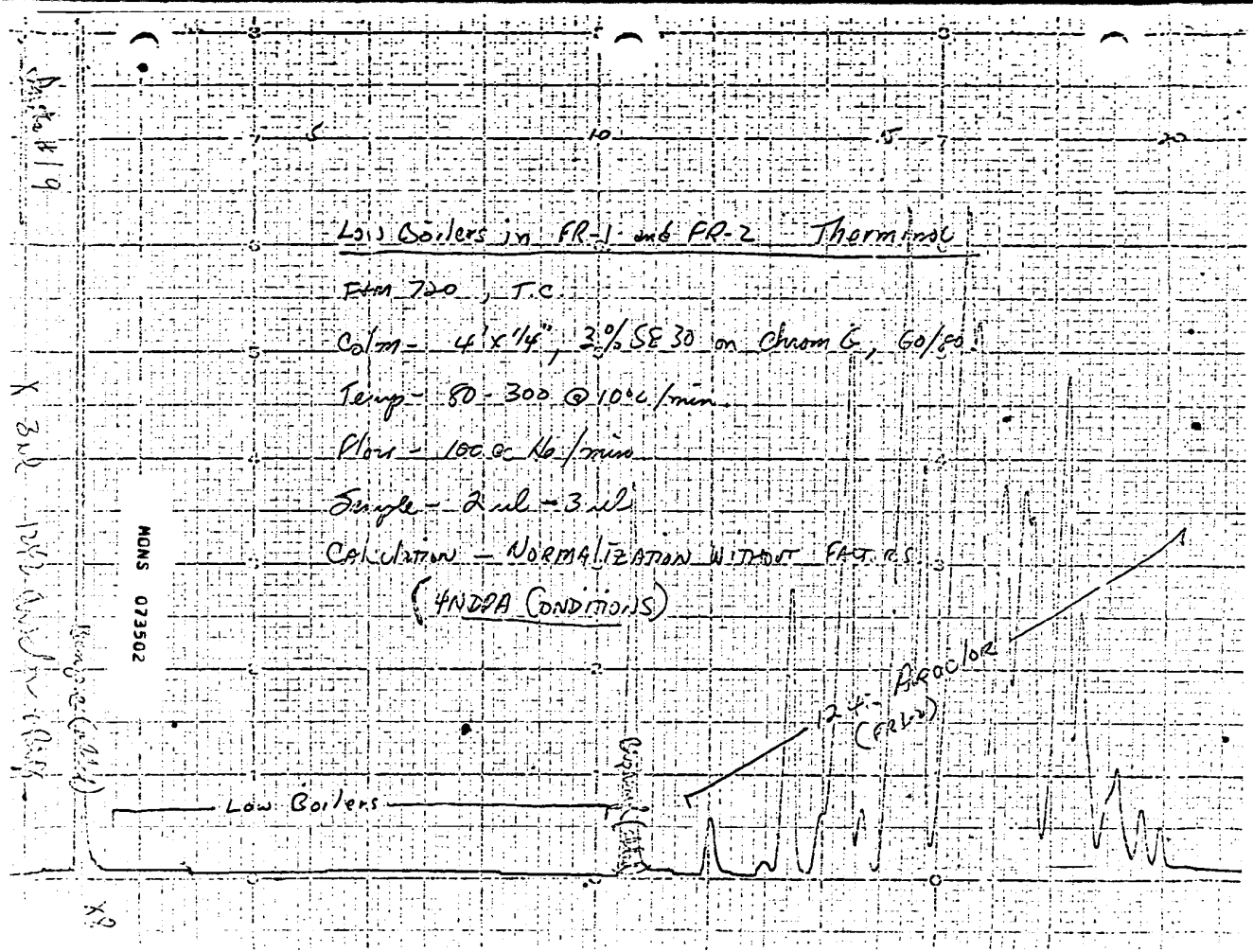
5. The low boilers elute between the benzene and biphenyl. Determine the counts in this area.

6. Compare the GLC curve to a standard curve and report extra peaks as "extra peak at____ minutes". (Exclude low boilers when reporting extra peaks.)

CALCULATION

$$\% \text{ Low Boilers} = \frac{\text{Counts for Low Boilers} \times 100}{\text{Total Counts}}$$

This is a normalized calculation and area correction factors and precision have not been determined. Reproducibility is expected to be ± 5% of the amount present.



Low Boilers in FR-1 and FR-2 Thermino
F&M 720, T.C.
Colm - 4' x 1/4", 3% SE 30 on Chrom G, 60/80
Temp - 80 - 300 @ 10°C/min
Flow - 100 cc He/min
Sample - 2 ul - 3 ul
Calculation - NORMALIZATION WITHOUT FACTORS
(4NDPA CONDITIONS)
124 - Aroclor
(FR1-2)
Low Boilers
MONS 073502