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Investigation of Laboratory Methods for the Determination of the lead content of fuels (part only)

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TRANSLATOR'S NOTE: This paper originally appeared as FKF Stuttgart Report 1382 - C.I.O.S. No. F.13, which has been translated in full. As published in the 1942 Year Book the paper contains in addition a discussion of a further method, the FKFS tri-chlor-acetic acid method: the relevant sections are translated and this document should be regarded as an appendix to document F.13.

II-9. FKFS Tri-chlor-acetic acid method (7)

(insert on p.8 of translation of F.13)

10 ccs. of the fuel to be tested are diluted with 10 ccs. of paraffinic gasoline. 1-2 grammes of solid tri-chlor-acetic acid are added to the gasoline mixture, and the whole is then refluxed in a 30 cc. flask with a standard ground glass joint to the condenser. When reaction is complete, the product is extracted three times with about 3 ccs. of water. The three aqueous extracts are drained off into a 100 cc. beaker through a separating funnel which is similarly attached to the boiling flask by a standard ground glass joint.

The liquid in the beaker is evaporated to dryness until white fumes of excess tri-chlor-acetic acid are given off. The tri-chlor-acetic acid is removed by heating carefully and not too strongly; this is best done by holding the beaker in a clamp, and keeping it in motion above the flame. A slight current of air above the beaker helps to disperse the fumes of the tri-chlor-acetic acid. 1 or 2 drops of ammonia are added to the residue obtained, and this is then brought to the boil with 2 to 3 ccs of 10% acetic acid and a little crystallized sodium acetate. The hot solution is treated with 5 ccs N/20 potassium dichromate, the lead being precipitated as lead chromate.

After boiling for a short time the lead chromate precipitate is rapidly cooled and filtered through a G.4 Gooch crucible into a 200 cc. Buchner flask. The filtrate is treated with about 0.2 grammes of potassium iodide and 5 ccs. of hydrochloric acid and back-titrated with N/20 sodium thio-sulphate solution. The number of ccs. of N/20 potassium di-chromate solution used multiplied by 0.0324, gives the T.E.L. content as percent by volume.

To carry out a check, the lead chromate precipitate in the filter can be weighed, and the volume of lead calculated.

IV-9 FKFS Tri-chlor-acetic acid method.

(insert on p.12 of translation of F.13)

To supplement the iodine method (6), which has already previously been used as a rapid method, the Research Institute for Transport and Automotive Engines at the Technical High School Stuttgart developed the tri-chlor-acetic acid method, which makes it possible to test fuels with a peroxide content and those rich in olefines, very accurately.

The T.E.L. is converted by the tri-chlor-acetic acid to lead chloride, with lead tri-ethyl tri-chloracetate and lead tri-ethyl chloride as intermediate stages. The lead in the lead chloride is estimated

as chromate. Tables 2 to 5 show that with all the fuels - even Rheinpreussen gasoline and Rumanian gasoline - the spread is comparatively small.

Table 2

FKFS Tri-chlor-acetic acid method

Lead determination in vol. % T.E.L. with a theoretical content of 0.0326 vol. %

Paraffinic gasoline	0.0328
Leuna gasoline	0.0330
Rheinpreussen gasoline	0.0327
Rumanian gasoline	0.0335
V.T. 702	0.0327
V.T. 707	0.0324
V.T. 810	0.0326
Limit of error (largest and smallest deviation from theoretical value, %)	(-0.06 (+2.80

Table 3

Lead determination in vol. % T.E.L. with a theoretical content of 0.0653 vol. %

Paraffinic gasoline	0.0655
Leuna gasoline	0.0656
Rheinpreussen gasoline	0.0656
Rumanian gasoline	0.0668
V.T. 702	0.0655
V.T. 707	0.0662
V.T. 810	0.0659
Limit of error (largest and smallest deviation from theoretical value, %)	(+0.3 (+2.45

Table 4

Lead determination in vol. % T.E.L. with a theoretical content of 0.1306 vol. %

Paraffinic gasoline	0.1294
Leuna gasoline	0.1311
Rheinpreussen gasoline	0.1315
Rumanian gasoline	0.1291
V.T. 702	0.1290
V.T. 707	0.1295
V.T. 810	0.1305
Limit of error (largest and smallest deviation from theoretical value, %	(-1.2 (+0.7

Table 5

Lead determination % T.E.L. in vol. for various aviation fuels

A_3 Aviation fuel	0.0555
B_4 Aviation fuel	0.1202
C_3 Aviation fuel	0.1210

Table 6

The apparent limits of error for the investigated lead methods and the necessary time for a lead determination

Method	Limit of error in % largest and smallest deviation from theoretical value of the mean of all leaded fuels	Period of time for a lead determination
FKFS Tri-chloracetic acid method	-0.5 to +1.98	30 mins.