SHORT COMMUNICATIONS

From a similar reduction of 5-trifluoromethyl-2,4-disulphamino-aniline were isolated 650 mg of a compound with an IR-spectrum identical with that of 5-methyl-2,4-disulphamino-aniline. (Found: C 31.78; H 4.22; N 15.92; S 24.02. Calc. for $C_4H_5N_3O_4S_2$: C 31.69; H 4.18; N 15.84; S 24.17.)


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Separation of Hg$^{++}$ from Active Carbon by Extraction with EDTA

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In the synthesis of vinyl chloride from acetylene and hydrogen chloride active carbon impregnated with mercuric chloride is used as a catalyst. The catalyst usually contains 8 - 10 % by wt. of mercuric chloride. The mercuric chloride content of catalyst samples drawn from a reactor which has been in use for a certain length of time, varies between zero and a value higher than the original one.

A producer of vinyl chloride is of course highly interested in a rapid and fairly accurate method for the determination of the mercury content of the catalyst. The present work was initiated some 20 months ago, at which time no publications dealing with this special problem could be found in available literature.

In our first experiments we investigated several procedures based on initial oxidation of the carbon, e.g. combustion in a Parr bomb, oxidation with fuming sulphuric acid and potassium persulphate and the method described by Schöniger$^{1,3}$ where the sample is burned in a conical flask filled with oxygen. By all these methods, however, the recovery of mercury was too low. This was also the case when the catalyst was extracted with acetone, while extraction with nitric acid or a mixture of nitric and hydrochloric acid seemed more promising. Another possible way of separating the mercury from the carbon may be distillation in a quartz apparatus, a method worked out for the determination of mercury in organic matter.$^4$

During our further work, however, we were led to the conclusion that the most convenient method would be direct extraction of the mercury by means of EDTA. Check analysis by the aqua regia/H$_2$O$_2$ extraction method described in Ref.$^4$ have shown that this extraction is complete also when applied on spent catalyst samples. In the extract the mercury content is easily determined photometrically as mercury dithizonate. For a more detailed description of the dithizone procedure, Ref.$^4$ may be consulted.

**Experimental.** All of the solutions are made up with glass distilled water.

Buffer solution, $pH = 10$. 70 g NH$_4$Cl and 610 ml 12N NH$_2$OH, diluted to 1000 ml.

EDTA-solution. Disodium salt of ethylene-diaminetetraacetic acid, 0.1 M.

Procedure. 20 - 30 mg of ground catalyst is transferred to a centrifuge tube together with 5 ml of EDTA-solution and 15 ml of buffer solution and extracted for 30 min with moderate shaking. The complexing of Hg$^{++}$ with EDTA is considered a very rapid reaction and it therefore seems probable that complete extraction is achieved in considerably less than 30 min. The extraction is followed by settling of the carbon, which is most conveniently done by means of centrifuging. 5 ml of the supernatant liquid is pipetted into a volumetric flask and the volume is adjusted to 100 ml. An aliquot of this solution is used for the subsequent determination with dithizone.


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