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THERMAL DECOMPOSITION OF INDUSTRIAL C_2Cl_6 CONTAINING SALTS

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ABSTRACT

The volatile and solid residues of the decomposed C_2Cl_6 of 98,0-99,8 % purity got from different suppliers were investigated by complex thermal methods /TG, DTG, DTA, MS/ and transmission IR spectroscopy. In this way compounds of very low concentration were determined: $CaCO_3$, Al_2O_3, SiO_2 and $Fe_4[Fe(CN)_6]_3$.

INTRODUCTION

In high quality Al semiproducts the maximum concentration of hydrogen, oxigen, sodium and calcium can be about 1 /ug/g. There are different molten metal treating processes to provide for this low impurity level: e.g. degassing with pure C_2Cl_6 salt. The aim of present work was to determine the decomposition products of the C_2Cl_6 after storage and heat treatment respectively.

MEASURING METHODS

TG, DTG, DTA curves were registered with a Mettler TA-1 thermoanalyser equipped with Balzers QMG 311 quadrupole mass spectrometer. Transmission IR spectra were taken with Zeiss UR-20 spectrophotometer.

The C_2Cl_6 samples were heated up to 330 and 800 ^oC in vacuum with a speed of 10 ^oC/min and mass spectra of the evolved gases were recorded simultaneously.

RESULTS AND DISCUSSION

The C_2Cl_6 sublimates without decomposition in air,

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causing loss of active material and enrichment of contaminants already at room temperature. The DTG, DTA curves of a partly sublimated blue-colorued sample can be seen in the Figure 1.

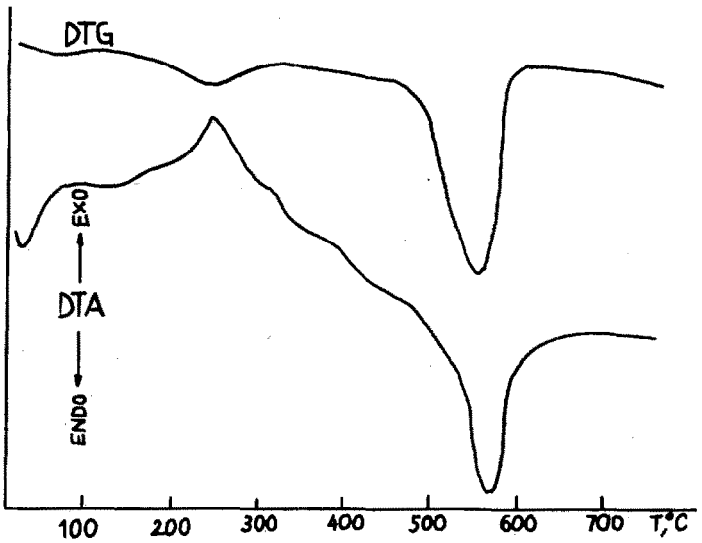

Figure 1. DTG, DTA curves of a partly sublimated blue-colorued C_2Cl_6 sample

The volatile decomposition products analysed by mass spectrometer are as follows: Cl containing ions below 200 °C; C and N ions between 200 - 500 °C and CO_2 above 500 °C /Figure 2./.

The IR spectra of the same sample before and after heating can be seen in the Figure 3. The results of the different methods, relating to anions, were in good agreement. The cations were determined with chemical analysis. Hence in the presented case the supposed thermal processes are: first C_2Cl_6 evaporates, then Fe-cyanocomplex decomposes and, at last, $CaCO_3$ depomposes.

By heating other pure C_2Cl_6 samples amorphous alumina and silicondioxyde contaminants were founded.

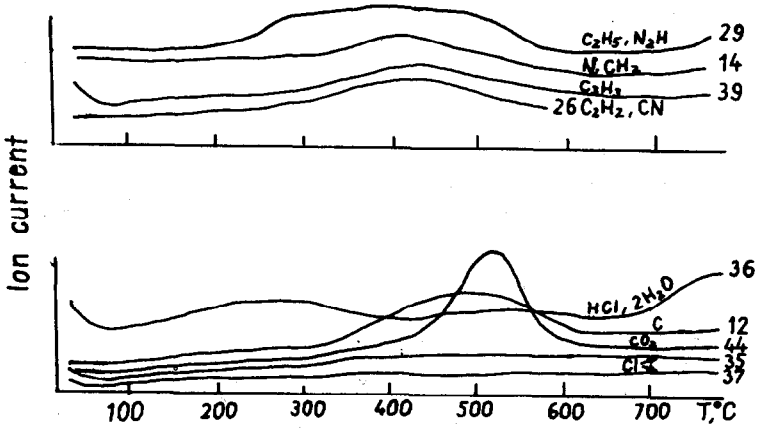

Figure 2. Ion current of different partial pressures in function of the temperature registrated by quadrupol mass spectrometer on the same sample as in Fig. 1.

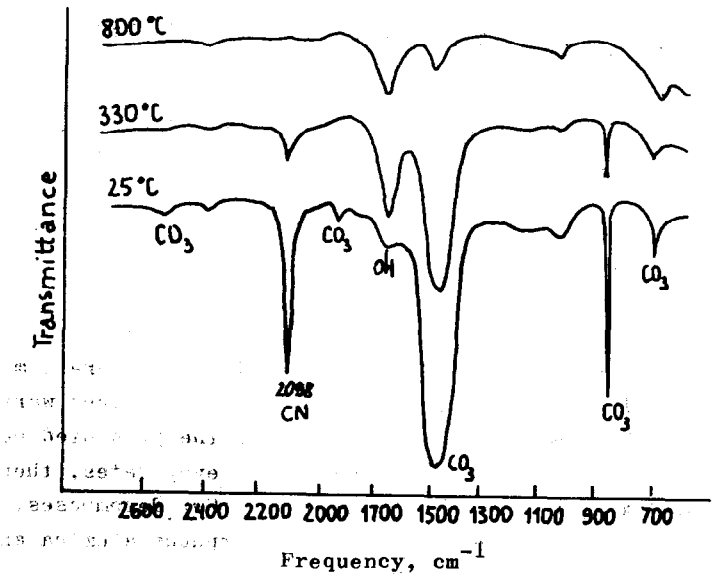

Figure 3. Infrared spectra of a partly sublimated blue-coloured sample heated at various temperature