

Thermal Analysis of the System: 1-Chloro-2,4-dinitrobenzene-Picryl Chloride

by

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Thermal analysis of the system: 1-chloro-2,4-dinitrobenzene-picryl chloride is of practical importance as the mixtures of the above compounds can be formed in the course of nitration of 1-chloro-2,4-dinitrobenzene to picryl chloride.

So far, existing data based on thermal analysis are controversial. According to Frankland and Garner [1], both compounds form an ordinary eutectic with freezing point 25.0°C. It contains 44% picryl chloride. Desvergnés [2] reported similar data: 25.6°C and 47.5%, respectively.

Jefremov and Frolova [3], however, discovered the existence of a molecular compound with a molecular ratio 1:1 containing 55% (by weight) of picryl chloride. The compound gives two eutectics: with freezing points 25.5° and 30.3°C containing 37% and 59% of picryl chloride, respectively.

In view of the existing controversy it appeared necessary to repeat the thermal analysis of the system in question once more.

Experimental

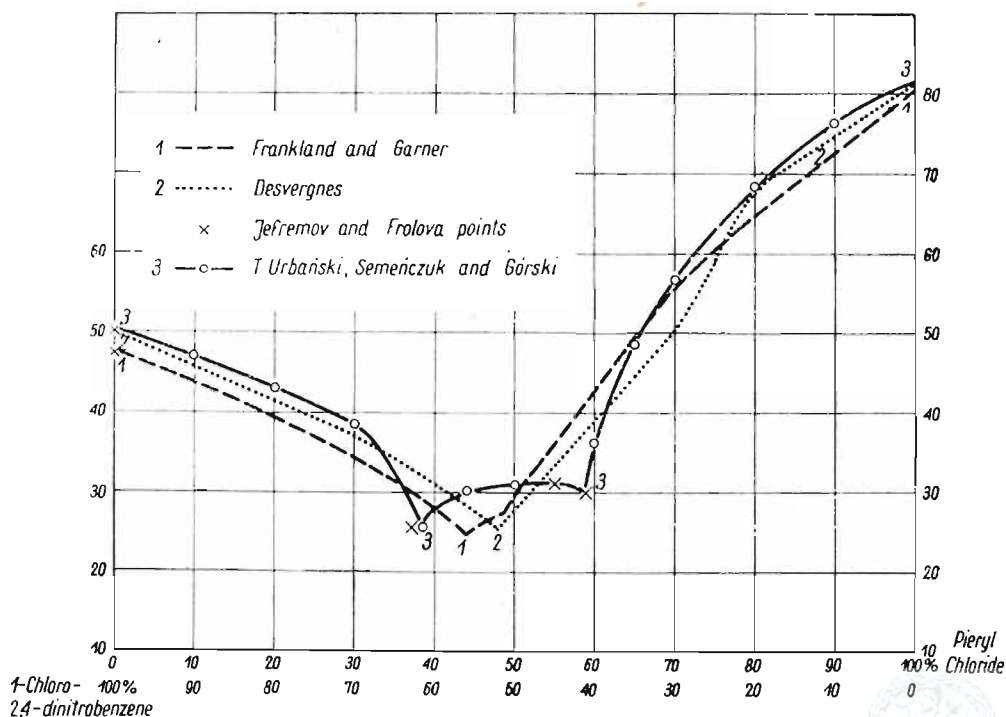
Picryl chloride was purified by crystallization of the commercial product using methanol as a solvent. Freezing p. 81.6°C was reached after repeated crystallizations.

1-Chloro-2,4-dinitrobenzene was prepared by nitration of *p*-chloronitrobenzene. The product was crystallized from ether, methanol and finally ether.

Thermal analysis was carried out in a conventional way by determining the beginning and end of freezing of the molten mixtures. Temperature was measured to 0.1°C.

The results are plotted in the Figure. Curves 1 and 2 correspond to the results of Frankland-Garner and Desvergnés, respectively. Those of our own experiments are depicted by curve 3. They are in good agreement with the results of Jefremov and Frolova.

The results of Jefremov and Frolova and ours are summarized in the Table.



TABLE

Authors	Eutectic 1		Compound		Eutectic 2	
	Picryl chloride content %	F. P. °C	Picryl chloride content %	F. P. °C	Picryl chloride content %	F. P. °C
Jefremov and Frolova	37	25.5	55	31.2	59	30.3
Urbański, Semeńczuk, Górski	38.5	25.5	55	31.15	59	30.2

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