DETECTION OF DINITROCELLULOSE IN PULPED NITROCELLULOSE BY X-RAY DIFFRACTION

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Nitrocellulose (containing about 13 per cent nitrogen) is the chief ingradient of single-base and double-base gun propellants. Dinitrocellulose in nitrocellulose has the effects of slowing down the burning rate and lowering the propelling power of nitorcellulose. As the X-ray diffraction pattern of pulped nitocellulose consists of diffuse bands, it is difficult to detect dinitrocellulose in pulped nitrocellulose by X-rays. If, however, the pulped nitrocellulose is kept for some time at liquid air temperature and the pattern is taken at room temperature, the photograph shows bands and sharp rings. The bands are of trinitrocellulose and rings are of dinitrocellulose. This is a method of detecting dinitrocellulose in pulped nitrocellulose.

Miles[1] has reported that the definition of X-ray diagrams of nitrocellulose improves with increase of nitrogen content from 10·5 to 12·8 per cent, until, when the critical point 12·8 per cent is passed, a definite pattern of cellulose trinitrate appears. Depending on the specimen the spots on the equitorial line spread into arcs and in case of pulped nitrocellulose one gets only diffuse bands. Between cellulose on one hand and trinitrocellulose on the other there is no product giving an X-ray diagram which is really characteristic. Further, he is of opinion that the grounds for regarding dinitrocellulose as a definite solid compound are quite insufficient. Happey[2], however, using highly resolved diagrams and oriented fibres of nitrocellulose has shown that (101) reflection of about 7 A may be a doublet consisting of spacings 6·9 A (dinitro form) and 7·25 A (trinitro form). He, therefore, thinks it is likely that nitrocellulose (with N. content between 12—14 per cent) is a two phase system containing tri and dinitro structures. According to him, the two phases can be distinguished by: (*a*) Spacing of 6·9 A and 4·0 A along the equator of the dinitro diagram and (*b*) a main spacing of 7·25 A (or in some cases 7·05 A) on the equator and a strong reflection of 4·45A on the first layer line and an obvious fibre diagram based on a repeat distance of 25·4 A along the fibre axis of the trinitro photograph.

X-ray photographs were taken at room temperature using Cu Kα (Ni filtered) radiation, 35 KV, 12 ma—exposure time 2 hours. After taking the pattern of nitrocellulose (13·1 per cent of N) the sample was sealed in an evacuated glass tube and kept immersed in liquid air for 5 hours. The tube was broken and X-ray pattern of the sample was taken at room temperature.

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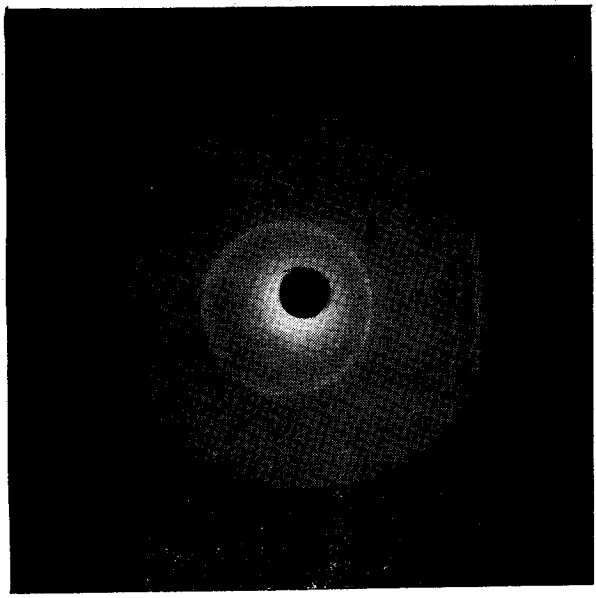

Fig. 1

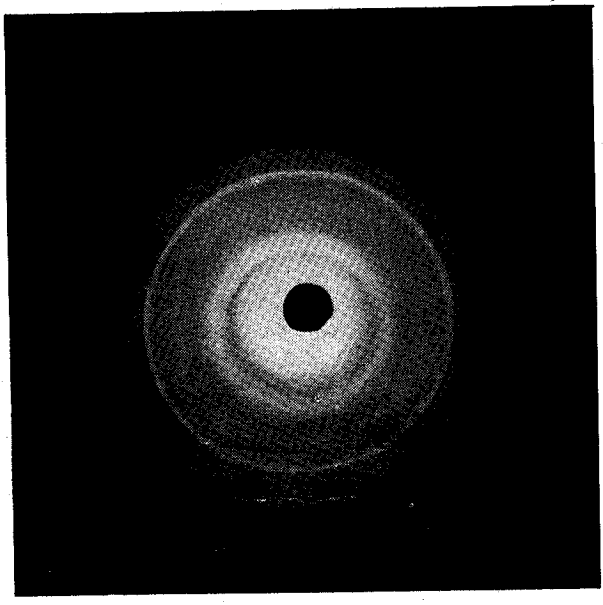

Fig. 2

X-ray diffraction patterns of Nitrocellulose (13·1% Nitrogen content).

1. Nitrocellulose not cooled.
2. Nitrocellulose cooled in liquid air.

Figures (1) and (2) are the patterns of uncooled and cooled specimens respectively. In case of uncooled specimen one inner (less diffuse) ring and two outer diffuse bands are obtained. The outer one of these bands is fainter. The pattern of the cooled specimen shows along with what just stated two extra sharp rings. (The inner most arc is from the collimator). 'd' values (in A) are given below:—

	Uncooled Sample	Cooled Sample
Inner ring ..	7·07	6·95
Diffuse band	4·92	4·79
Sharp ring	..	4·17
Sharp ring	..	3·70
Diffuse band	3·63	3·28

The 'd' values for the bands are not very accurate but these help to identify the pattern.

Referring to the criteria stated earlier, diffractions at 7·07 and 4·92 A correspond to trinitrate and that at 6·95 and 4·17 A to dinitrocellulose. The sample contained both dinitro and trinitro phases in amorphous form. When the sample is cooled the dinitro cellulose changes to crystalline form but the trinitrocellulose remains in amorphous form.

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References

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2. Happey, X-ray diffraction by polycrystalline materials—Peiser Rooksby, Wilson, The Institute of Physics, 539, 1955.