Notes

A SENSITIVE METHOD FOR DETECTING SUGARS ON PAPER CHROMATOGRAMS

Ek and Hultman[1] developed a colorimetric method for determining glucose in body fluids; they used the condensation reaction between the sugar and *p*-aminobenzoic acid or *m*-aminophenol in an almost water-free solution of glacial acetic acid to produce a coloured product. On the basis of their findings, an attempt was made in this laboratory to use a solution of *p*-aminobenzoic acid in glacial acetic acid for the identification of sugars separated by paper chromatography. The sugars used were raffinose, sucrose, lactose, glucose, galactose, fructose, mannose, xylose and rhamnose.

Development of the method

A 0·002-ml portion of a 0·2 per cent. solution, *i.e.*, 4 μg, of each sugar was placed on a piece of Whatman filter-paper, 10 inches × 10 inches, especially prepared for chromatographic work, the spots being about 3·5 cm from one of the edges of the paper and about 2·0 cm apart. A mixture of ethyl acetate, acetic acid and water (3:1:3) was used as developing solvent.[2] About 3 hours were needed for the solvent front to travel to the opposite edge of the paper when subjected to a descending-solvent technique. After completion of the run, the paper was dried for about 15 minutes in a current of air at room temperature; about 30° C. The dried paper was sprayed with a 1·0 per cent. solution of *p*-aminobenzoic acid in glacial acetic acid and heated at 105° C for about 10 minutes. Under these conditions, rather faint brownish spots for glucose, galactose and mannose and a distinct pinkish brown spot for xylose appeared on an extremely pale brownish yellow background; no spots were formed by the other sugars.

An attempt was then made to utilise the general principles of the chromatographic identification of sugars by converting the sugars to furfural or its derivatives and forming coloured products with *p*-aminobenzoic acid. As oxalic acid can be used to convert hexoses to ω-hydroxymethylfurfural[3], the use of this acid was tried. The spraying reagent could not be prepared by dissolving oxalic acid in a solution of *p*-aminobenzoic acid in *n*-butyl or *iso*butyl alcohol or any other common organic solvent, as an insoluble derivative appeared when the oxalic acid (0·5 g) was added to a 1 per cent. solution of *p*-aminobenzoic acid. A double-spraying technique was therefore used. After the chromatogram had been run and dried as before, it was uniformly sprayed with a 1 per cent. solution of *p*-aminobenzoic acid in *iso*butyl alcohol and dried at room temperature for about 15 minutes in a current of air; it was then sprayed with a 0·5 per cent. solution of oxalic acid in *iso*butyl alcohol and immediately heated at 105° C for about 10 minutes. In presence of 4-μg amounts of sugars, distinct chocolate-brown to yellowish brown spots on an

extremely pale greyish brown background were formed by glucose, galactose, mannose and xylose. Spots formed by sucrose, lactose and rhamnose were faint, and those formed by raffinose and fructose were extremely faint. In presence of 2-μg amounts of sugars, only glucose, galactose, mannose and xylose could be detected; the other sugars formed no spots.

However, when a 0·5 per cent. solution of oxalic acid in glacial acetic acid was used for the second spraying, 2-μg amounts of all the sugars formed easily detectable spots, although that formed by raffinose was somewhat faint; each spot had a sharp boundary. When an aniline - phthalic acid reagent[4] was used as spraying reagent and the amount of each sugar present was 4 μg, reasonably distinct spots were formed only by glucose, galactose, mannose and xylose, spots for the other sugars being either absent or extremely faint. Under identical conditions, spots developed with the proposed reagent were more distinct. When the amount of each sugar present was 1 μg, spots formed by glucose, galactose, mannose, xylose and rhamnose could be detected with the proposed reagent, whereas with the aniline - phthalic acid reagent, the only discernible spot was that formed by xylose.

A mixture of 2-μg amounts of raffinose, glucose, xylose and rhamnose, which have appreciably different R_F values in ethyl acetate - acetic acid - water mixture, was subjected to paper chromatography. Each of the separated sugars could be detected with the proposed spraying reagent; the spot formed by raffinose was extremely faint.

Conclusions

With 1-μg amounts of the sugars tested, the spots formed by glucose, mannose, xylose and rhamnose were faint but detectable. With 2-μg amounts, the intensity of the spots improved and with 4-μg amounts all spots were distinct. When the amount of each sugar present was increased to 8 μg (the largest amount tested), the spots were even more distinct.

The spots obtained by the proposed method are on an extremely pale greyish brown background and persist for several days without any appreciable reduction in colour intensity. The method is most sensitive and can be applied to a wide variety of sugars; it is hoped to apply it to the detection of various sugars in biological fluids, especially in human sweat.

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THE DETERMINATION OF GALLATES IN EDIBLE FATS

Under the Antioxidants in Food Regulations, 1958, limited amounts of *n*-propyl, *n*-octyl and *n*-dodecyl gallates are permitted in certain foods. To avoid the expenditure of unnecessary effort on samples eventually proving to be perfectly satisfactory, a rapid and reasonably accurate sorting test, with a recovery of at least 95 per cent., was desirable. It was considered sufficient to determine total gallate quantitatively.

Experimental

The normal colorimetric method for determining gallate, involving the use of iron, was unsatisfactory in methanolic solution. However, it was found that when a gallate in 95 per cent. methanol was shaken with solid ammonium ferrous sulphate, a clear and stable blue solution was obtained; such solutions obeyed Beer's law over the range 0·0 to 0·8 mg of gallate, maximum absorption in the visible region occurring at 5800 A. The absorption spectrum for *n*-propyl gallate is shown in Fig. 1; the shape of the curve is characteristic of all three gallates, but the optical density at any given concentration depends on the particular ester present.

Application of this reaction to extracts of fats in 95 per cent. methanol produced abnormal results, owing to the development of turbidity in the solutions and to the synergistic effects of other extracted matter. It was found that these effects could be nullified by shaking the extract with analytical-reagent grade calcium carbonate, which neutralised any extracted acidity and assisted in the clarification of the solution by coagulating some of the extraneous matter. The addition of 10 per cent. of analytical-reagent grade acetone after filtration further stabilised the solution, and recovery experiments showed that there was no detectable loss of gallate during treatment with calcium carbonate and that the colour was not affected (except by dilution) by addition of acetone. The use of solid ammonium ferrous sulphate removed those difficulties attendant on the use of an unstable reagent, such as ferrous tartrate solution.

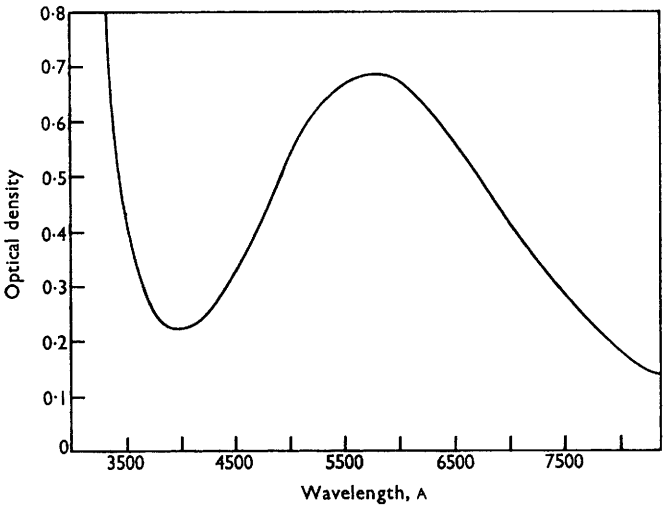

Fig. 1. Absorption spectrum of solution containing ammonium ferrous sulphate and 0·44 mg of *n*-propyl gallate in 95 per cent. methanol; final volume 11 ml

In a method described by Vos, Wessels and Six,[1] large volumes of fat were extracted with relatively small amounts of solvent; this necessitated several manipulations in order to achieve reasonable recovery. We have found that oils can be extracted directly with 95 per cent. methanol at 40° to 45° C and that solid fats can be extracted similarly when diluted with an equal volume of liquid paraffin. Under these conditions the recovery of added gallates in two extractions was 95 per cent.

Method

Apparatus—

Extraction vessels—The extraction vessels used consist of boiling tubes, each of which has a bulb of capacity 10 to 12 ml blown in its side at the bottom (see Fig. 2); they permit the separation of an upper layer simply by tilting the tube.

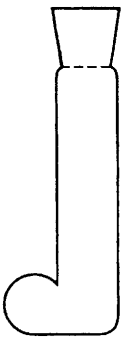

Fig. 2. Extraction vessel

Reagents—

Unless otherwise stated, all materials must be of recognised analytical grade.

Liquid paraffin—B.P. grade.
Methanol, 95 per cent.
Ammonium ferrous sulphate.
Calcium carbonate.
Acetone—Shake with calcium carbonate for 1 minute, and filter.

PROCEDURE—

Vigorously shake 10 g of warm liquid sample with 25 ml of 95 per cent. methanol for 1 minute in a vessel of the type shown in Fig. 2. (For solid samples, use a 5-g portion *plus* 5 ml of liquid paraffin.) Place in a water bath at 40° to 45° C, and allow to separate for about 15 minutes (separation into clear layers is unlikely and unnecessary). Pour the upper layer into a 50-ml calibrated flask, repeat the extraction with 20 ml of 95 per cent. methanol, again transfer the upper layer to the flask, and dilute to the mark. Add 1 g of calcium carbonate to the contents of the flask, shake for 30 seconds, filter through a Whatman No. 1 filter-paper, and reject the first few millilitres of filtrate. (The amount of calcium carbonate added is not critical, but must be sufficient to ensure a clear filtrate at this stage.) To 10 ml of filtrate add exactly 1 ml of acetone and about 10 mg of powdered ammonium ferrous sulphate, and shake for 1 minute. Set aside for 30 minutes to attain full colour development, and measure the optical density at 5800 A in 1-cm cells with a Unicam SP500 spectrophotometer. For extremely dilute solutions larger cells may be necessary.

Calculate the amount of gallate present per 11 ml of final solution from the expression—

$$\text{Gallate present, mg} = dk,$$

in which d is the optical density measured in 1-cm cells and k has the value 0·622, 0·785 or 0·952 for *n*-propyl, *n*-octyl or *n*-dodecyl gallate, respectively. (It is recommended that these factors be determined for each batch of reagents.)

RESULTS

The recoveries of gallates from various oils and fats by the proposed method are shown in Table I. When necessary, the esters can be distinguished from each other by a method such as that described by Vos, Wessels and Six.[1]

TABLE I

RECOVERY OF ADDED GALLATE FROM OILS AND FATS

Sample	Gallate added, p.p.m.	Recovery of— *n*-propyl gallate, %	*n*-octyl gallate, %	*n*-dodecyl gallate, %
Olive oil	100	95·7	96·2	95·4
	200	95·2	95·7	95·7
Lard	100	96·0	96·5	95·3
	200	96·2	96·0	95·8
Dripping	100	95·6	96·8	96·5
	200	96·1	96·3	95·7
Olive oil *plus* dripping	100	96·7	94·7	95·5
	200	95·9	95·5	96·1

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SPECIFIC MASKING BY ACETYLACETONE IN TITRATIONS WITH ETHYLENEDIAMINETETRA-ACETIC ACID

THE use of acetylacetone as a masking agent to improve the selectivity in potentiometric titrations with ethylenediaminetetra-acetic acid (EDTA) was first described by Fritz, Richard and Karraker.[1] During investigations into the application of certain masking agents in EDTA titrations with xylenol orange as indicator,[2] it was found that acetylacetone (2:4-pentanedione) could be used as a masking agent for aluminium, iron^{III}, beryllium, palladium and uranium when determining

zinc or lead. In Table I are summarised the masking effects of acetylacetone[1,3] on certain cations under the conditions used for titrating zinc or lead with xylenol orange as indicator.

TABLE I

MASKING EFFECT OF ACETYLACETONE ON CERTAIN CATIONS

Cations masked—

Fe^{3+}, Al^{3+}, Be^{2+}, Pd^{2+}, UO_2^{2+}

Cations partly masked—

Cu^{2+}, Hg^{2+}, Cr^{3+}, Ti^{4+}

Cations not masked—

Zn^{2+}, Pb^{2+}, Mn^{2+}, Ni^{2+}, Co^{2+}, Th^{4+}, Cd^{2+}, La^{3+}, Sn^{2+}, Bi^{3+}, Ce^{3+}

The complexes formed by acetylacetone with aluminium, iron, beryllium, palladium and uranium are much more stable than either the metal - xylenol orange complex or the metal - EDTA complex. Lead and zinc do not form stable complexes with acetylacetone and can therefore be titrated in the presence of any of the metals just mentioned without interference, provided that acetylacetone is added to the solution before titration. Zinc forms a weak complex with acetylacetone, especially in warm solution, but the zinc - EDTA complex is so strong that zinc can be removed from its complex with acetylacetone by titration with EDTA. Iron and uranium both produce somewhat intense colours with acetylacetone, and this makes normal titration and end-point detection impossible. The addition of an organic solvent, such as nitrobenzene or chlorobenzene, permits the colour to be extracted into the organic layer, and the end-point can then be detected in the aqueous layer.

Molybdate can also be masked with acetylacetone, but the complex so formed is stable only in strongly acid solution, and the reaction is of practical use only when titrating a metal such as bismuth.[2,4,5] In absence of acetylacetone, molybdate forms a weak complex with xylenol orange and also a precipitate with bismuth at the optimum pH for determining bismuth by titration against EDTA with xylenol orange as indicator.

TABLE II

EFFECTS OF VARIOUS SALTS ON TITRATION OF LEAD AND ZINC NITRATES WITH 0·1 *M* EDTA

Sample	Titre of 0·1 *M* EDTA for— solution containing zinc nitrate, ml	solution containing lead nitrate, ml
Solution A*	49·65	49·5
Solution B†	49·65	49·5
Solution B *plus* 1 g of $Fe(NO_3)_3.9H_2O$	49·7	49·55
Solution B *plus* 1 g of $Al(NO_3)_3.9H_2O$	49·65	49·5
Solution B *plus* 1 g of $UO_2(NO_3)_2.6H_2O$		
Solution B *plus* 1 g of $BeCO_3$ dissolved in nitric acid		
Solution B *plus* 1 g of $Pd(NO_3)_2$		

* Fifty millilitres of a solution containing zinc or lead, as nitrate.
† Solution A *plus* 5 ml of acetylacetone.

METHOD

PROCEDURE FOR DETERMINING ZINC OR LEAD—

In absence of other metals[2]—To 50 ml of a neutral or slightly acid solution of the sample add 2 ml of 5 *N* nitric acid and 5 g of hexamine or sufficient to produce a pH of 5 to 6. Titrate against 0·1 *M* EDTA with 5 drops of a 0·2 per cent. aqueous solution of xylenol orange as indicator until the colour changes from wine-red to pure yellow.

In presence of beryllium, palladium or aluminium—Proceed as described above, but add 5 ml of acetylacetone before the solution is buffered with hexamine. Acetylacetone forms an almost colourless insoluble precipitate with any of these metals.

In presence of iron or uranium—To 50 ml of a neutral or slightly acid solution of the sample in a stoppered 500-ml flask add 2 ml of 5 *N* nitric acid and a few drops of 20-volume hydrogen peroxide to oxidise any ferrous salt present. (In absence of iron this can be omitted.) To the

clear and cold solution add 5 ml of acetylacetone and then 5 g of hexamine to adjust the pH to between 5 and 6. Add 50 ml of nitrobenzene, and shake vigorously to extract the metal - acetylacetone complex into the organic layer. Dilute to 300 ml with water, add 1 ml of xylenol orange indicator solution, and titrate immediately with 0·1 *M* EDTA. Add titrant slowly as the end-point is approached, and observe the colour change in the aqueous layer.

The organic layer should not be removed, as it contains some zinc.

PROCEDURE FOR DETERMINING BISMUTH IN PRESENCE OF MOLYBDATE—

To the clear strongly acid solution containing bismuth and molybdate add 5 ml of acetylacetone. Mix well, and set aside until the acetylacetone - molybdate compound has been completely precipitated. Adjust the pH of the solution to between 1 and 1·5 with 5 *N* sodium hydroxide or 5 *N* ammonium hydroxide, add 1 ml of xylenol orange indicator solution, and titrate with 0·1 *M* EDTA. Towards the end of the titration, the colour of the solution begins to fade. At this point, add sufficient alkali dropwise to restore the red - orange colour of the bismuth - xylenol orange complex, and then complete the titration.

RESULTS

Table II shows the results found when solutions containing zinc nitrate and lead nitrate were titrated with 0·1 *M* EDTA in the presence of acetylacetone and various metallic salts.

We thank the Directors of Hopkin and Williams Ltd. for permission to publish this Note.

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DETERMINATION OF SULPHIDE SULPHUR IN MINERALS

A RAPID and accurate method for determining sulphide sulphur in minerals containing the sulphides of lead, zinc, bismuth, manganese, nickel, cobalt, silver, etc., has been described.[1] This method consisted in reducing the mineral with hydriodic acid to evolve hydrogen sulphide, which was swept off in a current of hydrogen or nitrogen and absorbed by a suspension of cadmium hydroxide, the sulphide being determined iodimetrically. It was pointed out, however, that this method could not be applied to the analysis of pyrite and chalcopyrite, as the reaction is extremely slow.

We have found that the reaction can be hastened to completion by using a more concentrated solution of hydriodic acid (analytical-reagent grade, sp.gr. 1·7, containing from 54 to 56 per cent. of hydrogen iodide). Solution of such minerals as pyrite or chalcopyrite is smooth and complete, provided that a pellet of mercury is added to the sample. It is necessary to heat the contents of the reaction vessel gently. The free iodine usually present in the hydriodic acid can easily be reduced by adding a few crystals of sodium hypophosphite; this gives a colourless reagent solution.

METHOD

PROCEDURE—

Place 50 to 100 mg of powdered mineral (it need not necessarily be finely pulverised) in the reaction vessel,[1] add a small pellet of mercury, and displace the air in the apparatus by hydrogen. Add about 5 ml of hydriodic acid, and gently warm the flask. Sweep off the hydrogen sulphide, absorb it in an alkaline suspension of cadmium hydroxide, and carry out the determination as described previously.[1] The entire procedure can be carried out in 1 hour.

After removal of hydrogen iodide by boiling with concentrated sulphuric acid, the residue in the reaction vessel can be used for determining other components of the sample by standard methods.

RESULTS

Some representative samples of sulphide minerals were analysed by the proposed method; the results are shown in Table I.

TABLE I

SULPHIDE SULPHUR FOUND IN VARIOUS MINERALS

Mineral	Sulphur content of pure mineral, %	Sulphur found, %
Iron pyrites	53·42	52·16
Chalcopyrite	34·89	33·83
Stibnite	28·32	28·30
Realgar	29·93	29·11
Orpiment	39·05	38·36
Sphalerite	32·86	31·65

It can be seen that the sulphide sulphur present can be determined with reasonable accuracy The results of duplicate determinations agreed to within 0·5 per cent., and determinations of total sulphur by wet oxidation gave the same values as those found by the proposed method, provided that the mineral had not undergone any appreciable oxidation.

DISCUSSION OF THE METHOD

In view of the commercial and practical importance of determining sulphur in such mine as pyrite, the proposed method provides a valuable and rapid procedure. There is no possib of any interference by other radicles, such as iron, and no danger of contamination by the prod of combustion when gas flames are used (these combustion products usually contain oxides sulphur, which are reported to be taken up avidly by alkali fluxes and oxidising solutions).[2]

The preparation of a solution in determining the sulphur content of antimony sulphide minerals is said to be difficult,[3] but a naturally occurring sample of stibnite can be analysed for sulphur in 1 hour by the proposed method; the result obtained agrees with the calculated value This is also true for arsenic sulphide minerals.

If pyrite is treated with concentrated hydriodic acid alone, about 14 per cent. of the sulphur present is evolved as hydrogen sulphide, whereas, in presence of mercury, the entire amount is recovered as hydrogen sulphide. In presence of mercury, even a more dilute reagent solution such as that prepared by mixing concentrated hydrochloric acid and potassium iodide solution, will react with pyrite to evolve hydrogen sulphide quantitatively. By virtue of its reducing action and ability to form complexes with metallic derivatives and so hold them in solution hydrogen iodide can react with such a mineral as pyrite, which is insoluble in a non-oxidisin acid, *e.g.*, concentrated hydrochloric acid.[4]

Solution of pyrite in presence of mercury is due to galvanic effects rather than to chem reaction. The reaction is slow when a mercury salt (mercuric chloride) is used instead of met mercury. Any metal more electropositive than iron, *e.g.*, lead, tin, copper or silver in powd form, will have the same effect as mercury, whereas zinc and aluminium, which are more el negative than iron, will not. All these metals specified dissolve rapidly in the acid, but, m is more electropositive than most of them and is easy to handle; its use is therefore pref Further work is in progress to ascertain the role of mercury and other metals in the solu pyrite by hydriodic acid.

We thank Professor M. R. A. Rao for his valuable suggestions and keen interest in this

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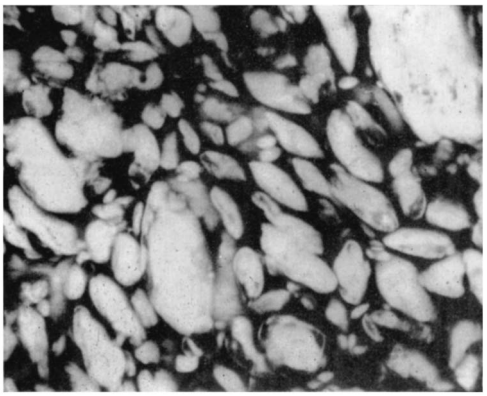
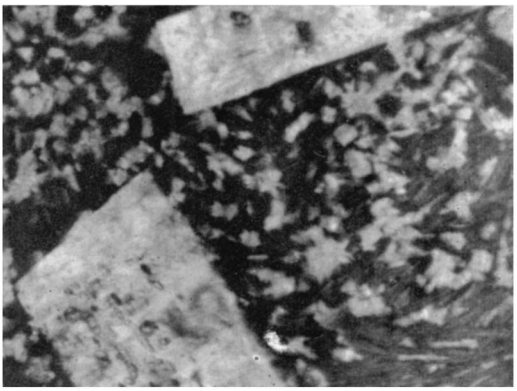
THE STAINING OF CAST HIGH EXPLOSIVES FOR OBSERVATION OF THE CRYSTALLINE STRUCTURE

THE nature of the microstructure of cast high explosives is of importance, since in some insta it can be correlated with the explosive properties.[1,2,3] Various observational techniques been described.



(*a*)

(*b*)



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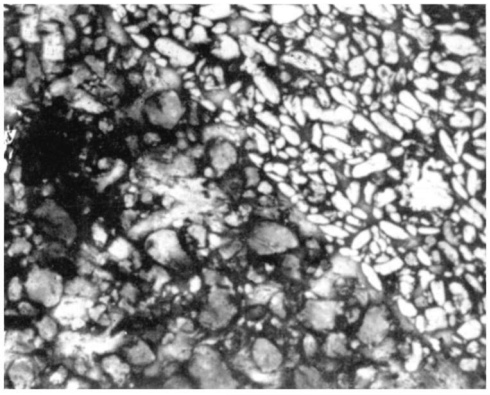

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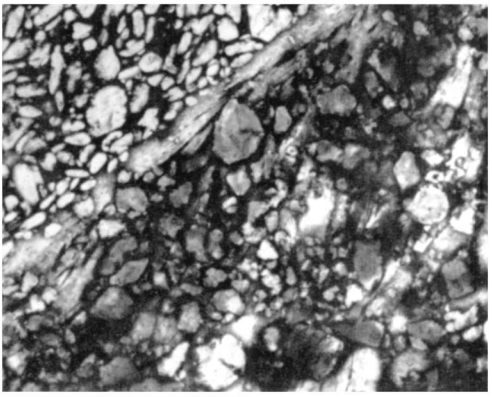

(*e*)

Fig. 1. Typical photomicrographs: (*a*) RDX in matrix of TNT; (*b*) PETN in matrix of TNT; (*c*) 60/40 tetryl - TNT; (*d*) and (*e*) boundary between 60/40 RDX - TNT and 50/50 amatol

[To face p. 300

Williamson[2,4] has used two methods, a microtome technique in which a thin film of explosive adhering to a backing material is examined under a polarising microscope and a "film and cast" technique in which a cast of the surface features is made and studied. Another approach is to use a stain to show up one component. Pristera[5] has investigated the reactions between trinitrotoluene (TNT) and the hydroxides of sodium, potassium and ammonium, which produce a reddish colour.[6,7] He found that *N* potassium hydroxide in diethylene glycol can be successfully used as a staining agent on an explosive specimen cooled by solid carbon dioxide.

A more convenient staining technique can be based on the coloured complexes formed between some nitrobodies and organic bases.[8] The most convenient base for this purpose has been found to be dimethylaniline, which gives a red colour with TNT, orange with tetryl and picric acid and no colour with the other common high explosives. The method used is to cut a section about 3 mm thick from the end of a casting by means of a fine saw and to smooth one face of this by rubbing gently on glass-paper. The specimen is then glued by this face to a microscope slide and its thickness reduced to about 1 mm with a microtome. The surface is lightly wiped first with a cotton-wool pad moistened with acetone and then one wetted with dimethylaniline. The specimen is examined under a microscope by transmitted white light.

A selection of typical photomicrographs obtained by this method is shown in Fig. 1. Figs. 1 (*a*) and 1 (*b*) show *symo*-trimethylene trinitramine (RDX) and pentaerythritol tetranitrate (PETN), respectively, in a matrix of TNT. Fig. 1 (*c*) shows the structure of 60/40 tetryl - TNT, in which the crystals of tetryl are embedded in a matrix of the eutectic conglomerate.

In investigations of the transition of detonation from one explosive to another good contact between the two compositions is essential, and this can best be done by casting one material on to the other.[9] Figs. 1 (*d*) and 1 (*e*) show about 5 mm of the boundary between 60/40 RDX - TNT and 50/50 amatol. Fig. 1 (*d*) shows a perfect join, but Fig. 1 (*e*) shows what happens when there is a wide temperature difference between the already solidified substance and the second melt. A surface layer rich in TNT is formed,[3] and this can be seen along the interface.

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