

Slurry and Emulsion Explosives: New Tools for Terrorists, New Challenges for Detection and Identification.

Charles R. Midkiff Jr. and Allan N. Walters

*Forensic Science Laboratory, National Laboratory Center, BATF Rockville, Maryland and
U.S. Postal Service Laboratory, Washington, DC respectively.*

Abstract

With advantages in cost, safety and ease of use, water gel/slurry and emulsion explosives are rapidly replacing traditional explosives. As a result, cap sensitive versions of each are increasingly encountered in criminal activity and a strategy for their identification and characterization is needed. A water gel/slurry explosive is essentially an aqueous solution of an inorganic oxidizer gelled with a carbonaceous gelling agent. Dispersion of fuel or additional oxidizer in the gel produces a slurry, the "water gel explosive" of commerce. Added sensitizers enhance initiation to a detonator with particular compounds protected by patent and specific to a producer. When intact material is available for examination, sensitizer and other component identification characterizes the explosive type. Emulsion explosives, more efficient than gels, differ in that, emulsifying agents suspend droplets of aqueous oxidizer solution in an oil phase. Chemical sensitizers may be used in emulsion explosives but are less common than in slurry types. Most emulsions are sensitized by microspheres, tiny glass bubbles which both control density and provide "hot spot" initiation. Physical characteristics and composition of typical slurries and emulsions, combined with a systematic analytical approach, are used for the characterization and discrimination of intact gel/slurry and emulsion explosives.

Introduction

For reasons of safety, storage and economics, in the past two decades, commercial makers and users of explosives have moved away from traditional high explosives such as dynamite. As a result of decreased use of this once widely available explosive, the explosives used in criminal and terrorist incidents are changing. In the early 1970's, the principal explosive encountered in the ATF laboratories was commercial dynamite [1]. While dynamite is now far less frequent in criminal bombings in the United States, its commercial replacement, the slurry or emulsion explosive, is increasingly being encountered as criminals become familiar with these newer types of high explosives.

In North America, and to an increasing extent worldwide, for large scale commercial blasting, mining, construction etc., the major volume high explosives today represent a radical departure from traditional explosives' concepts. Whereas water represented a problem to users of dynamites and blasting agents such as ANFO, its successors have water as a significant component.

Gel or Slurry Explosives

A more recent explosive, resembling a gelatin dessert, is referred to as either a water gel or slurry. Rigorously, most are slurries because they contain one or more solid components suspended in a continuous semi-solid or gel phase. No clearcut distinction between gels and slurries appears to be made within the explosives industry and for convenience, the terms will be considered synonymous. These explosives consist essentially of a water solution of an inorganic oxidizer such as ammonium nitrate, or for better density control or sensitivity, mixtures of ammonium with sodium or calcium nitrate, gelled with a natural polysaccharide such as guar gum. Additional crystalline oxidizer and fuel is suspended in the gel matrix. Other ingredients may be added as fuels, sensitizers, stabilizers, etc. Common components of water gel/slurry type explosives are listed below.

Table 1

Ingredients for Slurry Explosives

Ingredient	Function		Ingredient	Function
NH ₄ NO ₃	Oxidizer		Guar gum	Gelling Agent
NaNO ₃	"		Starch	"
Ca (NO ₃) ₂	Oxid./Density		KCrO ₄	cross-linking
NaClO ₄	Oxid./Sens.		Sb comp'd	"
Microspheres	Density/Sens.		Bi "	"
Amine nitrate	Sensitizer		Boric Acid	"
Glycol nitrate	"		Borax	"
flake TNT	"		Aluminum	Fuel/Sensit.
Smokeless pwd	"			

Additional explosive and non-explosive components considered for use in slurry explosives are listed by Sudweeks [2] and Kaye [3].

A more modern, but related, water containing explosive, the emulsion, differs from the gels in that emulsions consist of two distinct phases, an oil phase and a water phase. These too, are increasingly becoming tools for criminal or terrorist activity and their characterization will be discussed subsequently.

Slurry/gel or emulsion explosives capable of direct initiation by a number 8 detonator or blasting cap are classified as cap sensitive and subject to rigorous handling and storage requirements. Slurry/emulsion explosives not initiated by a No. 8 cap, are classified as blasting agents and subject to less restrictive regulation. These require a primer for initiation but, once initiated, usually have detonation velocities in the high explosive range. Blasting

agents are infrequently encountered in our laboratory but can be characterized using the approach to be described.

Cap sensitive slurries were first used for commercial blasting in Canada about 1957 [4] but about two years earlier, water gel explosives sensitized with N,N-bis (trinitroethyl) urea and capable of initiation by a No. 8 detonator had been developed in Sweden [5]. Initially, North American slurry explosives were sensitized with high explosives such as TNT, low explosives such as smokeless powder or with flake aluminum [6]. A variety of other materials, both explosive and non-explosive, were proposed and/or used by explosive producers as slurry sensitizers during the 1960's but most were not completely satisfactory. Although attractive for a variety of applications, early gels had a number of limitations. Among these was the tendency of ingredients to segregate at elevated temperatures or for the oxidizer to salt out at low temperatures, rendering the explosive ineffective. Addition of surfactants and use of calcium nitrate as a co-oxidizer improved low temperature sensitivity. Cross-linking of the gelling agent with boron, antimony or bismuth compounds produced a gel of improved stability and largely solved both segregation and salting out problems [7]. A second major advance in water gel explosives was the introduction in the mid 1960's of sensitizers such as nitrate salts of hexamine, ethanolamine or monomethylamine, although at the levels used to achieve adequate sensitivity, they should be considered as significant components in the final product. Because each sensitizer is covered by patent, identification of the sensitizer compound, if any, is an effective method for identification of the producer of a gel explosive. **Table 2** contains a representative listing of sensitizers for slurry explosives, including some no longer used but which could be encountered in old products.

Table 2

Chemical Sensitizers for Slurry Type Explosives

Company	Brand Name	Sensitizer	Ref.
Dupont/ETI	Tovex	Mono Methylamine Nitrate	26
Hercules	Flogel	Smokeless powder (DNT optional)	31
Hercules/IRECO	IREGEL	Mono Ethanolamine Nitrate	27
Thermex/SEC	Detagel	Hexamine Nitrate	28
IRECO	IREGEL-435	Aluminum	3
C.I.L.	Powermex	Ethylene glycol mononitrate	19
Trojan/IMC	Trojel	Nitrostarch	3
ICI/Nobel	Supergex	Isopropyl nitrate	29

In addition to chemical sensitizers, slurry/emulsion type explosives are also sensitized by voids in the form of tiny bubbles. Air may be entrained at the time of mixing or bubbles produced by reaction of additives. Reproducible entrainment; however, is difficult and entrained gas is less effective as a sensitizer at low temperatures. In addition, problems arise when the explosive is subject to hydrostatic pressures, as for example, a column of cartridges loaded vertically in a borehole. Current practice relies on incorporation of hollow glass, resin or ceramic spheres known as microspheres or microballoons. These micron-size spheres serve two functions in explosives; they may be added to control density, critical for detonation wave propagation, or function as either a primary or secondary sensitizer. Glass spheres are transparent, resin spheres translucent and ceramic types are opaque. With few makers of microspheres and different types of glass used, producers are distinguishable. This information can be of value when a rigorous comparison is required, as for example, comparing a sample obtained from a suspect with a sample of known source. While microspheres have other applications, for example, as a filler in epoxy potting compounds, their wide use in slurry/emulsion explosives is a useful indicator that the material being examined is one of these.

Most major explosives producers consider the formulations of slurry or gel explosives proprietary so little compositional information is found in the literature to aid the analyst in characterization of these materials. Information on ingredients and expected concentration ranges is best obtained from the original patents. Kaye [3] has an extensive listing of slurry explosive related patents with patent title, number, and name of company to whom assigned but its coverage is only prior to 1980. One of the authors (CRM) is preparing a listing of patents dealing with slurry and emulsion explosives to expand the coverage to more recent products which, when completed, will be available on request. For illustration purposes, **Table 3** shows nominal compositions, obtained from published information, for several former and current commercial gel/slurry explosives.

Cap sensitive slurry type explosives are generally packaged in plastic bags or tubes and resemble commercial sausage packaging when intact. Cartridges range in diameter from 1 to 4 inches and 8 to 16 inches in length. Polyethylene, polyvinyl chloride and polyester films are used for packaging and the tubes may be clamped at each end with a small metal band. For former DuPont products (now made by ETI), a blue colored sleeve indicates a permissible explosive, i.e. one approved for use in underground coal mines.

Emulsion Explosives

One of the newer commercial explosive developments, competing with both commercial dynamite and the water gel slurries is the cap sensitive emulsion. In these water-in-oil emulsions, a droplet of a supersaturated solution of ammonium nitrate is surrounded by a hydrocarbon serving as the fuel. A typical emulsion consists of water, one or more inorganic nitrate oxidizers, oil (with or without dissolved wax) and emulsifying agents of the water-in-oil type. Emulsions may contain chemical sensitizers such as metal perchlorates to improve initiation at low temperatures but often rely on microspheres for sensitization as well as density control. The size of the spheres exerts a significant effect on the sensitivity and detonation velocity of the explosive [8-9]. In a study of emulsions prepared with sieved

Table 3

Nominal Composition of Sensitized Slurries

Product	Producer	NH ₄ NO ₃	NaNO ₃	Sensitizer	H ₂ O/gum/Al	Misc
Hydromex	C.I.L.	38%	25%	25% TNT	12%	
DBA-1	IRECO	50	15	25 TNT	10	
Tovex 300	Dupont/ETI	30.8	14	36.7 MMAN	19.5	
Tovex 800	"	43	8.5	20 "	29.5	
SSS	"	18	6.7	35.3 "	22.1	10*
TR-2	"	23.5	13.2	30 "	32.3	
CS Booster	Hercules	38.2	-	33.5 EGMN	18.6/1.0/-	#

* Calcium nitrate

Ethylene glycol/crosslinking agent/microspheres (4.2/0.5/4.0)

spheres, velocities obtained with 64 μm spheres were considerably higher than with those of 82, 108, 111 and 153 μm and velocity fall off with increasing density was much less severe [10]. Several years ago, it was indicated that "All emulsions manufactured at Atlas now use bubbles or microspheres for sensitizers" [11], however, our laboratory has also identified perchlorates in Atlas Powermax formulations so the possibility of additional sensitizers in an emulsion should not be overlooked. Additional samples will be examined to determine if secondary sensitizers are a standard ingredient for a particular product or are added, as needed, for improved low temperature sensitivity to ensure reliable initiation in cold climates. The consistency of most emulsions resembles that of cream cheese and they may or may not be aluminized. In general, the aluminum in emulsions is discrete granules, as compared to the fine paint-grade or atomized material used to increase the energy of slurries. Representative ingredients of emulsion explosives are shown in Table 4.

Until recent years, Atlas enforced its basic patents [12-13] and was the only U.S. domestic producer of emulsions. Because emulsions release 93% of their calculated thermochemical energy versus 50-70% for gels, they are extremely attractive on a cost/performance basis and, in addition, exhibit excellent storage stability. As a result, since expiration of the Atlas patent, emulsion explosives have become available from most major manufacturers and numerous patents on emulsion explosives worldwide have been located in a survey of the *Chemical Abstracts* database. Emulsions may be packaged in plain Kraft, waxed or plastic coated paper cartridges closely resembling commercial dynamite. Package sizes range from 1 to 3 1/2 inches in diameter and 8-16 inches long. An interesting difference between

Table 4

Ingredients for Emulsion Explosives

Ingredient	Function		Ingredient	Function
NH ₄ NO ₃	Oxidizer		Paraffin Oil	Fuel
NaNO ₃	"		Microchrystl. Wax	"
Ca (NO ₃) ₂	Oxid./Density		Paraffin Wax	"
NaClO ₄	Oxid./Sens.		Aluminum, granular	"
Microspheres	Density/Sens		Sorbitol esters	Emulsif
Amine nitrate	Sensitizer		Sorbitan esters	"
Diesel fuel	Fuel		Oleates, various	"
Tall Oil	"		Ethanolamides	"

permissible emulsions and dynamites is that the dynamite usually contains several percent of sodium chloride to suppress flash whereas sodium chloride may not be needed in the permissible emulsion. One example of a permissible emulsion is Atlas 7D.

A variety of two component emulsification systems have been described in patents for emulsion explosives. Despite the usage level, usually less than 2%, of these in the finished product, identification of the emulsifiers is an area for further study. Particular combinations, as for example, Sorbitan sesquioleate and oleic diethanolamide, may be consistently used by a manufacturer and serve as a guide to producer identification.

Combinations of oxidizers are frequent in emulsions and to further reduce dust initiation in mines, emulsions have been made with complete replacement of the ammonium nitrate component by sodium or calcium nitrate [14]. Although compositionally quite similar, there are a range of emulsion formulations, depending upon desired performance. Nominal compositions of several emulsions are shown in **Table 5**. For these, except as indicated, the formulation is for the basic emulsion and microspheres are added to obtain the desired density.

Laboratory Examination

When a sample of a suspected water gel slurry or emulsion is received in the laboratory, visual examination provides an indication of its type. Typically, water gels have a consistency resembling that of set gelatin or gel-type toothpaste and range from nearly clear, often with suspended white particles, to an off white or cream color. Aluminized slurries are silver grey. Emulsions are frequently off white in color and may contain visible aluminum

Table 5

Nominal Composition of Emulsion Explosives

NH ₄ NO ₃	NaNO ₃	H ₂ O	Emulsifier	Oil/Wax	Oxidiz.	Ref
78.7%	-	16.0	1.5	3.8	-	9
66.91	-	12	1.5	5.0	14.59 ^a	10
78.9	-	15.8	1.5 ^b	3.8 ^b	-	8
57.8	17.4	15	1.5/0.6	4.3	-	23
64.22	2.85	11.4	0.95	2.85/0.95	9.88 ^c	13
67.6	3.0	14.0	1.0	1.0/3.0	10.4 ^d	13

a Calcium nitrate

b Estimated (total oil and emulsifier 5.3%)

c Sodium perchlorate; plus Aluminum 5.00% & glass bubbles 2.0%

d Sodium perchlorate; two types of wax - 1.5% of each

granules. For initial examination, a small portion of the original sample is placed directly on a microscope slide and examined at 30-40X. Observation of small spheres, often of different sizes and resembling bubbles, suggests a slurry or emulsion explosive. In addition, shiny particles of aluminum or grains of inorganic nitrates may also be visible. Several grams of the slurry or emulsion sample in a vial is shaken with 2-3 mL of hexane or pentane. The water gel or slurry will be essentially unchanged but the emulsion will disintegrate immediately. If the sample is tentatively identified as a gel or slurry, a few grams of the sample are diluted with water and placed in an ultrasonic bath for about 15 minutes to disrupt the gel structure or, alternatively, a small homogenizer can be used. Ten percent nitric acid has been suggested for destruction of the gel structure [15] but in our laboratory, for qualitative purposes, has not been found necessary. The sample is filtered or, more satisfactorily, centrifuged and the aqueous filtrate removed. When centrifugation is used, intact microspheres float on the surface and are collected using a pipette. They are deposited on a glass slide for optical microscopy or on a stub for examination by Scanning Electron Microscopy/ Energy Dispersive X-Ray (SEM/EDX) or alternatively, by X-ray Fluorescence. In the U.S., there are two major manufacturers of hollow glass microspheres, differing in composition, allowing the producers to be distinguished. This information is useful in sample comparisons. **Table 6** shows elemental analysis results for several types of microspheres.

The aqueous layer is removed and tested for nitrate with the modified Griess test and for ammonium with Nessler reagent. Testing for chloride with silver nitrate is useful because NaCl may be used in a permissible formulation or to increase density. A flame test will identify sodium and, with a cobalt glass, potassium, if present. If a small spectroscope is available, characteristic calcium lines may be observed with formulations containing calcium nitrate. Ion chromatography (IC) of the aqueous extract readily detects

Table 6

Elemental Analysis of Microspheres

<i>Producer</i>	<i>Type</i>	<i>Brand Name</i>	<i>Elements by SEM/EDX</i>
3M	glass	Scotchlite B23/500	Ca, Si, B*
PQ	glass	Q-Cell	Na, Si
PQ	ceramic	Extendspheres SG	Al, Ca, Fe, K, S, Si, Ti
Unknown	phenolic	unknown	Al, Cl, I, Na, S, Si
Unknown	glass	from Hercules	Al, Ca, Cl, K, S, Si

*with light element detector

ammonium, sodium, calcium, nitrate, and monomethylamine [15]. Although not essential with bulk samples, IC is convenient and routinely used in our laboratory. Ammonium and sodium nitrate can be identified when a portion of the aqueous extract is evaporated to dryness and examined by X-ray diffraction. Examination of a portion of the filtrate by AA or ICP spectrometry may detect antimony or bismuth, compounds of which are used in cross-linking the gel but has not been evaluated in our laboratory. Similarly, boron compounds may be detectable by addition of methanol to the filtrate and passage of the vapor, containing the volatile methyl borate into a flame.

If they have not previously been removed, the residue from the water extraction can be examined microscopically for microspheres. When suspected aluminum particles are observed, they are tested on a microscope slide with one drop of 10% NaOH. A separate portion is tested with 6N HCl. Aluminum reacts readily with NaOH to release bubbles and HCl reacts after an induction period. For definitive comparison of two samples, the morphology of the aluminum particles can be examined on the electron microscope.

Amine salts used as sensitizers are detected in the water extract. Parker [16] has described a modified spot test for primary and secondary amines to detect monomethylamine nitrate (MMAN) with a limit of detection of about 20ug and reports that ammonium nitrate does not interfere. Parker also describes a TLC method for MMAN using cellulose plates and a chloroform/methanol/water eluent. Visualization is based on detection of the nitrate salt with oversprays of diphenylamine (DPA), UV light and H₂SO₄ with an LOD of 0.5ug. Three different TLC systems for MMAN and monoethanolamine nitrate (MEAN) on cellulose or silica gel separate the amine nitrates from sodium and ammonium nitrate [17]. Eluent systems were chloroform/methanol/water, chloroform/methanol and chloroform/ethanol/water/HCl. Visualization was by overspray with ninhydrin and heating to develop the color or by the UV fluorescence of the derivative formed with the amine and fluoescamine. A less expensive alternative to fluoescamine, which we plan to evaluate, is o-phthaldehyde.

More convenient than testing the filtrate is direct extraction of a portion of the intact slurry with methanol, which, while dissolving some ammonium nitrate, in TLC, is easier to work with than an aqueous extract. A cellulose plate (Avicel F, Analtech Inc. or equivalent) is used with a solvent of $\text{CHCl}_3/\text{CH}_3\text{OH}/\text{H}_2\text{O}$ (50:45:7) and initially developed with an overspray of 0.15% ninhydrin in freon. Heating of the plate visualizes the amine salt as rose-red but the spot frequently exhibits streaking. The plate is then sprayed with Diphenylamine in ethanol and exposed to long wave UV. Both the amine spot and that from the ammonium nitrate appear as lavender spots with NH_4NO_3 having a R_f in this system of about 0.19.

Although we have examined a number of TLC systems, varying plate type and mobile phase, further work is needed to improve spot structure, obtain a R_f higher than 0.34 for MMAN, and to evaluate the TLC behavior of other amine nitrate sensitizers. Spray reagents giving different colors with amino acids of different structure have been reported and merit evaluation.

Another approach to the detection of MMAN is extraction of the salt, formation of a TNBS [18] or dansyl derivative and identification by high-performance liquid chromatography (HPLC) using UV detection at 254 nm. With this approach, MMAN was detected in a water extract of a fragment of plastic film wrapper from Tovex [19]. We have similarly identified MMAN in an empty Tovex wrapper by swabbing with methanol and examining the extract by TLC. Prime and Krebs [19] detected the sensitizer, ethylene glycol mononitrate, in debris from explosion of the water gel slurry, Powermex. A purge-and-trap collection procedure on charcoal, and direct injection on the HPLC was used. Sensitive detection of MMAN and MEAN using HPLC with photolysis and electrochemical detection has also been reported [20]. In this work, a water extract containing the sensitizer is derivatized with 2,4-dinitrofluorobenzene and the effluent from the HPLC examined with and without photolysis prior to electrochemical detection. The amine derivative is detectable only in the "lamp-on" position of the photolysis unit. In IC analysis, addition of methanol to the mobile phase is reported to improve separation for MMA and monoethanolamine [21] but poses problems with swelling of some types of ion-exchange resins. A method for quantitative IC analysis of the amines and nitrates in water gel explosives is used by an explosives producer for routine quality control [15]. IC also permitted the determination of the relative concentrations of Na^+ , NH_4^+ , K^+ and MMA to distinguish two apparently similar water gels [22].

For emulsions, the hexane or pentane extract is centrifuged and microspheres on the surface removed for examination as previously described. The initial organic extract is removed and a second extraction performed to ensure complete removal of the hydrocarbon phase. The extracts are combined and filtered through a 45 micron filter to remove suspended particulates. The extract is evaporated to a small volume by warming in a water bath and the residual oil/wax examined by high temperature gas chromatography. Although useful for sample comparisons, the value of characterization of the oil or wax as indicative of a particular producer is unknown because material from different suppliers may be used by a single emulsion producer [23] and a number of different trade named oils have been mentioned in the patents issued on these materials.

The residue remaining after the organic extraction is dried, then extracted with hot water. Aluminum granules remain after hot water extraction and are identified using instrumental or chemical tests. The aqueous extract is tested for NH_4^+ , Na^+ , Ca^{++} , Cl^- and NO_3^- using

the chemical and instrumental tests previously described. In addition, spot or crystal tests for perchlorates using methylene blue/ $ZnSO_4$, triphenylselenium chloride or tetrabutyl ammonium chloride should be conducted to identify this sensitizer, if present. Depending on concentration, a lavender precipitate or violet needles are formed with methylene blue, a white precipitate or white needles in clusters with triphenyl selenium chloride and tetrabutyl ammonium chloride forms a white precipitate or small crystals resembling chips of glass. Alternatively, IC for anions and cations can be used. Although amine-type chemical sensitizers are infrequent in emulsions, one which has been used in some formulations is ethylenediamine dinitrate. Formation of a complex with zinc permits determination of ethylenediamine by IC at levels of about 1 ppm without interference from other monovalent cations and amines [24].

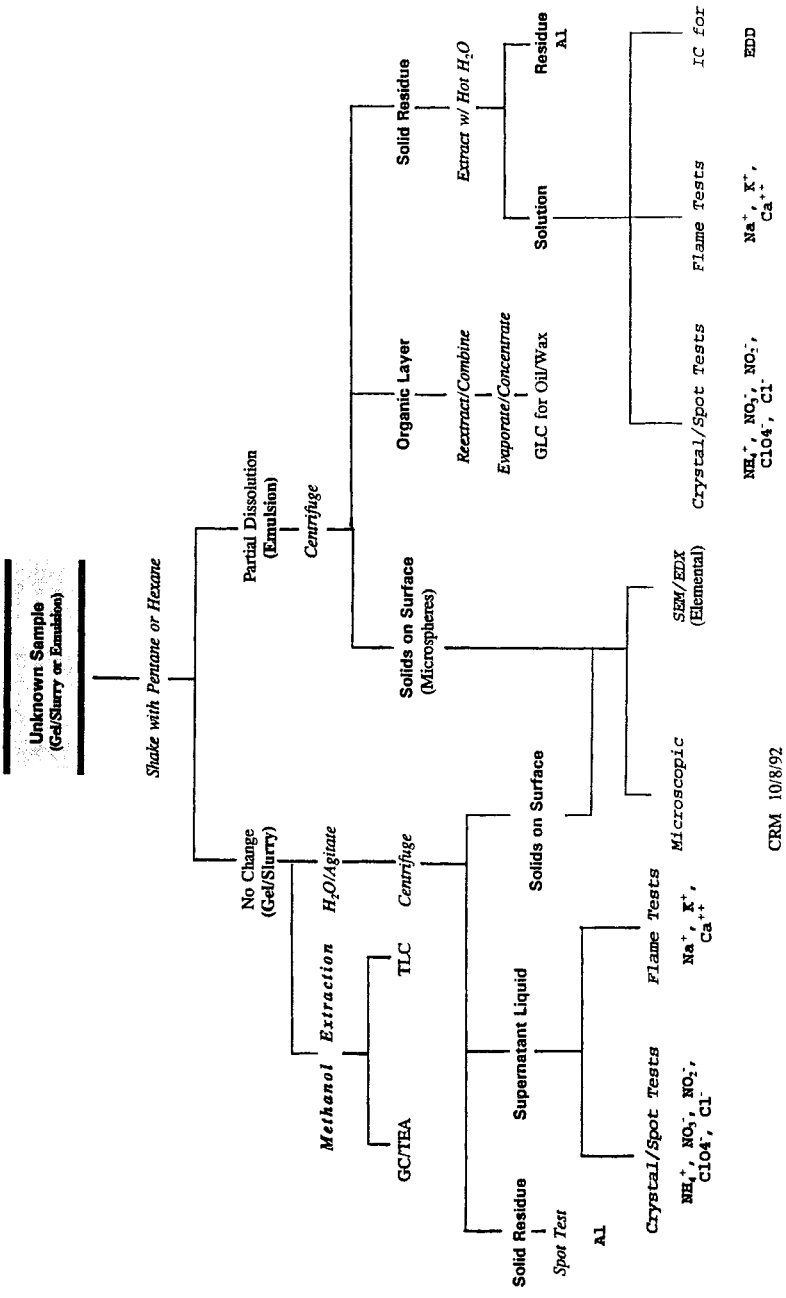
Conclusion

Cap-sensitive gels, slurries and emulsions are rapidly replacing more traditional commercial explosives. They are increasingly becoming available to, and employed by, terrorists and criminals. Identification and characterization of these explosives by the laboratory is essential to effectively assist in the investigation and prosecution of bombing incidents. The following flow chart is an expanded version of an analytical scheme for the examination of slurry and emulsion explosives [25]. Using a series of relatively simple chemical and instrumental tests, samples of only a few grams of an intact slurry or emulsion explosive can be identified by type and characterized to the extent required. With a systematic approach, these explosives, new tools for terrorists, become less of a challenge for identification and characterization in the forensic laboratory.

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Analytical Scheme



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