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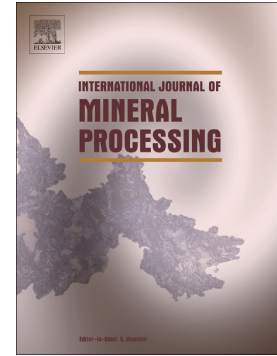
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Sodium peroxide fusion for reliable determination of gold in ores and metallurgical samples

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Abstract

A protocol based on peroxide fusion has been developed for the determination of Au in metallurgical samples and ores covering a wide range of concentrations and diverse mineralogies. The quantity of Na_2O_2 required was minimised, while maintaining lixiviant efficacy, by the use of mixed $\text{NaOH} + \text{Na}_2\text{O}_2$ fluxes. The method was shown to be applicable to the determination of coarsely-particulate gold. Fusion cakes were dissolved in hydrochloric acid and then extracted using methylisobutylketone to provide concentrations appropriate for quantification by graphite furnace atomic absorption spectroscopy and to minimise interferences from dissolved salts. The protocol was tested on a variety of certified reference materials and produced results in excellent agreement (at Au concentrations ranging from 0.05 to 47 $\mu\text{g g}^{-1}$) with the traditional lead fire assay method, while avoiding the health and safety issues associated with that technique.

Keywords

Gold, peroxide fusion, metallurgical samples, ores

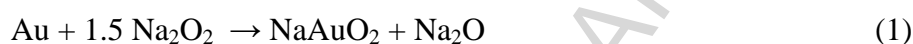
1. Introduction

In commercial mineral assaying the determination of gold in ores and industrial mineral processing streams is a significant practical challenge. The occurrence of gold as ductile macroparticles of $>100 \mu\text{m}$ diameter in some samples is particularly problematic. Such particles are not, in general, reduced in size or homogenised with the more brittle mineral matrix of the sample by commonly employed preparation techniques such as fine grinding (“pulverisation”) with vibratory mills.^{1,2} To achieve reproducible analytical results for such materials it is usually necessary to employ large subsamples, typically 25 to 50 g.^{3,4} Dissolution and extraction of Au from these samples inevitably requires large amounts of reagents, and generates considerable quantities of waste, with attendant occupational safety and health (OSH) concerns.

The standard analytical technique used by the minerals industry for geological resource definition and for operational purposes is fire assay using lead collection (PbFA).^{5,6} This technique involves decomposition

of the sample by fusion with sodium carbonate + sodium tetraborate mixtures in the presence of appropriate amounts of lead(II) oxide, PbO. Unlike other common techniques for Au determination, such as aqua regia digestion⁵ and fast cyanidation (e.g LeachWELL™),^{3,9} PbFA is able to fully decompose all sample types, with complete liberation of Au, and readily accommodates subsample weights up to 50 g. However, PbFA is accompanied by significant occupational safety and health (OSH)⁷ and waste disposal⁸ concerns, which arise mostly from the large amounts of PbO required: up to tonnes per month in large commercial laboratories. Reagentless options such as neutron⁹ and gamma¹⁰ activation analyses have been used to quantify gold directly in ores but their application in commercial mineral assay laboratories is seldom practicable.

Two major alternatives to ensure complete sample decomposition for Au determinations in mineral and metallurgical samples have been developed. Acid digestions incorporating hydrofluoric acid (HF)¹¹ completely dissolve gold-bearing silicate, oxide and sulphide minerals. However, the use of HF in quantities sufficient to digest 25 to 50 g samples is costly, has serious OSH implications, and creates a problem of fluoride waste disposal. The second option is to use an oxidizing, alkaline, sodium peroxide fusion (PF) or sinter.¹²



This typically involves decomposing samples using 4 to 10 times their weight of sodium peroxide (Na₂O₂) at 500 to 700 °C.¹² Upon cooling, the fusion cake obtained is brought into solution for analysis using sequential reaction with water and hydrochloric acid (HCl). The PF approach has three distinct advantages: first, PF decomposes all minerals so that many elements, in addition to Au, can be determined from a single digestion; second, there are minimal OSH concerns; and third, the waste generated is mostly sodium chloride.

Although the PF approach has been used to determine Au in geochemical research samples of up to 20 g, commercial mineral assay laboratories have chosen not to adopt it due to the expense of the Na₂O₂ required for the large samples required for heterogeneous ores.^{13,14} However, the cost of such analyses may be lowered by the addition of less expensive reagents.¹³ For example, radiochemical analyses have employed Na₂O₂ + sodium hydroxide fusion mixtures containing ~30 % (w/w) NaOH,¹⁵⁻¹⁹ albeit using sample sizes of < 1 g. Sodium carbonate (Na₂CO₃) has also been proposed as an additive²⁰ but few details regarding the efficacy of such mixes are available.

A second difficulty with PF is that it introduces large amounts of salts into the analyte solution. This creates problems for quantification when using graphite furnace atomic absorption spectroscopy (GF-AAS) and especially inductively coupled plasma mass spectrometry (ICP-MS), making additional sample clean-up procedures necessary in order to generate reliable data. No single approach to address this problem has proven to be fully satisfactory. For example, use of ion exchange (IX) resins resulted in

erratic or low Au recoveries.²¹⁻²³ Separation of Au from salts by reductive co-precipitation with tellurium is effective^{13,14,24} but too complex for routine commercial work. Mention has been made of Au recovery from PFs by sorption on polyurethane but no substantive details provided.²⁵ A more promising approach is offered by solvent extraction (SX), e.g., with methylisobutylketone (MIBK), which is known to be effective in extracting Au from aqua regia digest solutions.^{26,27} The separation of Au from dissolved salts has the added advantage of providing a concentrating step, which can be important when measuring Au concentrations in analyte solutions at the sub- $\mu\text{g L}^{-1}$ level.

Given the positive benefits of PF for whole sample dissolution, and the significant OSH and waste difficulties associated with the current PbFA method, a PF-based protocol for commercial laboratory Au analysis is potentially attractive. The present work details the development and validation of a PF protocol that uses significantly less Na_2O_2 in the fusion step, has a simple acid leach (AL) dissolution step, and a robust solvent extraction step. The resulting procedure is suitable for the routine determination of Au in metallurgical samples and ores over a wide range of Au concentration and sample mineralogy.

2. Experimental

2.1 Reagents and reference materials

Analytical grade NaOH (Ajax, Australia >97.5 %), Na_2CO_3 (Ajax, >99.8 %), Na_2O_2 (Rowe Scientific, Australia, >93 %) and HCl (Ajax, 32%), technical grade methylisobutylketone (MIBK) (Rowe, >99 %), precipitated silica (Ajax, >98%), and 99.99% purity Au wire (0.5 or 1 mm diameter) were used without further purification.

Specimens of metallic Au were flattened to $90 \pm 10 \mu\text{m}$ by hammering Au wire between hardened steel surfaces to mimic the shape of macroparticulate Au in ground ore samples. The flattened specimens were then boiled in concentrated HCl, rinsed with deionised water, and cut to size and weighed ($\pm 1 \mu\text{g}$) using a Sartorius Model 4503 microbalance. Dissolution experiments used 1 mm diameter Au wire without treatment.

All solutions were prepared gravimetrically to avoid the uncertainties associated with volumetric procedures. A standard solution ($7.746 \mu\text{g-Au g}^{-1}$) for GF-AAS analyses was prepared gravimetrically by dissolving 7.394 mg of Au in 10 mL of aqua regia, evaporating to near dryness and diluting with 2 M HCl.

Certified reference materials (CRMs, Table 1) were obtained from Ore Research and Exploration Pty Ltd (Melbourne, Australia), and used without further treatment as per the supplier's recommendations.

Table 1: Certified Reference Material descriptions and selected compositional data. Full details can be found at the supplier's web site.³⁷

Certified Reference Material	Description			
		SiO_2 (%)	Fe (%)	S (%)

OREAS 24b	Granodiorite Lithogeochem / Blank	66	4	<1
OREAS 45e	Lateritic Soil Lithogeochem	40	24	<1
OREAS 901	Cu-Au ore	75 ^b	4	<1
OREAS 504b	Porphyry Cu-Au-Mo Volcanic Hosted Massive Sulphide	60 ^b	7	1
OREAS 622	Zn-Pb-Cu-Ag-Au Ore	54	4	8
OREAS 210	Au Ore	na.	9 ^c	3
OREAS 62e	Au-Ag Ore	66	3	<1
OREAS 208	Au Ore	46	15	5
OREAS 991	Cu-Au-Ag Concentrate ^a	20 ^b	27	31

^a Contains 21% Cu

^b Not provided in the certificate of analysis. Estimated by difference using other available compositional data

^c Aqua regia extractable. Total may be significantly higher.

2.2 Dissolution of metallic Au in Na₂O₂ melts

Mixtures (total mass 5 g) of Na₂O₂, NaOH and, where present, Na₂CO₃ in varying ratios were weighed into a zirconium crucible and melted with agitation for five minutes in a 55 mm i.d. tube furnace at 640 °C. The crucible was briefly withdrawn from the furnace to add a 1 mm diameter Au wire specimen. The melt was re-heated at 640 °C without agitation for 15 minutes, withdrawn from the furnace, and allowed to cool for several minutes until solid. After dissolution of the fusion cake in water the bright Au wire was recovered, rinsed with deionised water, dried, and re-weighed. The extent of Au dissolution was determined by mass loss and converted to thickness loss (Δd) assuming the Au wire to be a cylinder of uniform density 19.3 g cm⁻³ and negligible change in length:

$$\Delta d = 2\sqrt{(\pi\sigma l)^{-1}} (\sqrt{m_1} - \sqrt{m_2})$$

where σ is the density, l the length (constant), m_1 the original mass, and m_2 the mass of the wire after reaction. Reproducibility was typically $\pm 5\%$ relative, over the range of Na₂O₂ concentrations investigated.

2.3 Leaching recoveries

Flattened Au specimens (~400 μ g, the minimum required for convenient manipulation) were fused with 1 g of NaOH + 1 g Na₂O₂ in Zr crucibles for 15-20 minutes in a muffle furnace at 640 °C. The cooled crucible and its contents were immersed in 50 mL of either water or 2 M HCl in a borosilicate beaker at room temperature, swirled gently until the cake had completely dissolved, and then diluted to 100 mL with 2 M HCl.

2.4 Optimised protocol for Au determination in certified reference materials

Mixtures of 0.25 g certified reference material + 1 g each of NaOH and Na₂O₂ were heated in Zr crucibles at 640 °C for 15 minutes with occasional swirling. Cooled crucibles were immersed in 25 mL of 2 M HCl in a borosilicate beaker and periodically swirled for up to an hour until the cake dissolved. Solutions were diluted gravimetrically with 2 M HCl in polyethylene screwcap sample bottles to give 70 to 100 g of fusion-digest solution, and allowed to stand overnight. To ensure that the final concentration of samples fell within the GFAAS calibration range, weighed aliquots of 0.1 to 8 g of the digest solutions and 1.6 g of MIBK were transferred to 10 mL flat-bottomed polypropylene screwcap tubes. Samples were made up to 10 mL with 1 M NaCl + 1 M HCl solution, mixed by inversion for two minutes, and centrifuged for up to 15 minutes. The upper (MIBK) layer was removed and analysed by GF-AAS.

2.5 Analysis by GF-AAS

GF-AAS was performed with a GBC XplorAA double beam instrument equipped with deuterium-lamp background correction, a GF3000 graphite furnace atomisation system and a PAL 3000 auto sampler. The graphite furnace program was adapted from Branch *et al.*,²⁶ with additional optimisation of the ashing temperature and the sample injection volume (Table 2). Signal smoothing was used to optimise baseline noise and peak height reproducibility. The effect of solvent evaporation from the open autosampler thimbles²⁷ was corrected for by appropriate interpolations based on the measurement of bracketed calibration solutions.

Table 2: GFAAS settings

Parameter	Setting			
Wavelength	242.8 nm			
Lamp current	4 mA			
2D lamp background correction	On			
Slit width	1.0 nm			
Slit height reduction option	On			
Measurement mode	Peak height			
Signal smoothing	"Time constant" setting 0.5 s			
Purge gas	Nitrogen (> 99.99 % purity)			
Sample injection volume	30 µL			
Replicates	3			
Furnace program	Final T (°C)	Ramp time to T (s)	Hold time at T (s)	Purge gas
Cooling delay before injection	20	-	30	on
Dry	150	10	-	on
Ash 1	450	20	-	on
Ash 2	700	20	-	on
Atomise	2200	1	4	off
Tube clean	2400	1	3	on

The instrumental LOD for Au in the MIBK solution, calculated as three standard deviations of the Au concentration measured for MIBK blanks, was 0.1 ng-Au (g-MIBK solution)⁻¹, in accord with previous reports.²⁷ Relative standard deviations (RSD) for replicate atomisations of the same solution were generally $\leq 1\%$; duplicate solutions generally gave RSDs $\leq 2\%$.

3. Results and discussion

3.1 Dissolution of metallic Au in Na₂O₂-based melts

Au in commercial mineral samples is often macroparticulate in nature.³ However, very little information on the department of macroparticulate Au in PFs is available in the open literature.^{28,29} Almost all previous studies on the determination of Au using PFs^{13,20,21} have utilized small samples (< 1 g) in which the Au was in nano-particulate form and thus homogeneously distributed. Accordingly, it was considered appropriate to investigate PF conditions suitable for the easy dissolution of the relatively large Au macroparticles found in typical ore samples. This included an exploration of the extent to which Na₂O₂ could be diluted with the less expensive NaOH and/or Na₂CO₃ while retaining its efficacy for Au dissolution. As no CRMs containing macro-particulate Au were available, measurement of the dissolution of elemental Au in Na₂O₂ melts was performed using gold wire.

Used alone, Na₂O₂ sintered but did not fully melt at 640 °C, making it difficult to immerse the Au wire and to measure its dissolution; full melts were achieved with the addition of $\geq 10\%$ w/w of NaOH. The dissolution of metallic Au in Na₂O₂ + NaOH mixtures was found to be maximal at 50:50 w/w (Fig. 1) but decreased steadily at higher NaOH concentrations. However, even mixtures containing ca. 80 % (w/w) NaOH showed greater dissolution of Au than those containing 10 % NaOH. In addition, the data indicated that Au macroparticles, which are typically flattened to thicknesses of < 20 μm by conventional laboratory pulverisation,³⁰ should dissolve fully in Na₂O₂ + NaOH mixtures with Na₂O₂ concentrations as low as 10 % w/w. This offers significant cost savings for routine Au determinations by PF. Costs could be further reduced by the use of ‘synthesis’ or ‘industrial’ grade Na₂O₂ but these have not been tested here due to resource limitations.

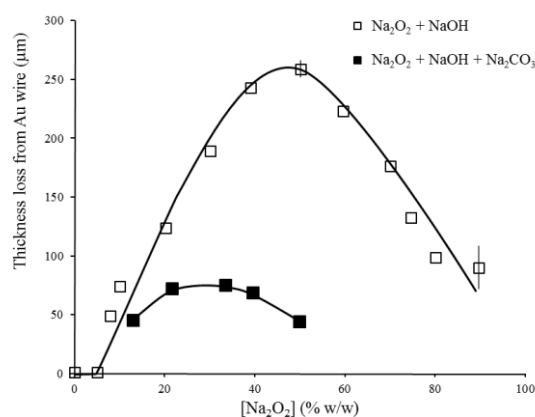


Figure 1: Extent of dissolution of Au wire in unstirred binary ($\text{Na}_2\text{O}_2 + \text{NaOH}$) or ternary ($\text{Na}_2\text{O}_2 + \text{NaOH} + 50\% \text{ w/w Na}_2\text{CO}_3$) melts held at 640°C for 15 minutes

The success of the $\text{NaOH} + \text{Na}_2\text{O}_2$ mixtures in dissolving metallic Au prompted investigation of the addition of Na_2CO_3 , given that Totland *et al.*²⁰ reported using $\text{Na}_2\text{O}_2 + \text{Na}_2\text{CO}_3$ mixtures to decompose sample residues not amenable to pressurized HF acid digestion. Sodium carbonate is less deliquescent than NaOH and it was thought that addition of Na_2CO_3 would result in PF mixtures that were easier to handle. However, all mixtures of $\text{Na}_2\text{O}_2 + \text{Na}_2\text{CO}_3$ tested produced particularly viscous melts at 640°C that were difficult to work with and thus were not considered viable for routine assay work. Ternary (x:50:y) mixtures of $\text{NaOH} + \text{Na}_2\text{CO}_3 + \text{Na}_2\text{O}_2$ resulted in better melt behaviour but proved to be much less effective than the binary $\text{NaOH} + \text{Na}_2\text{O}_2$ mixtures (Fig. 1). Thus, all further work employed the latter mixtures.

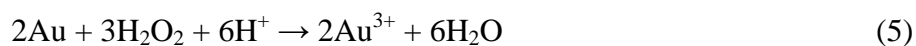
3.2 Leaching of fusion cakes

Reaction of the PF cake with water would be expected to produce NaOH and sparingly-soluble brown $\text{Au}(\text{OH})_3$ from the hydrolysis of Na_2O and NaAuO_2 , eqn (2), respectively. However, the considerable stoichiometric excess of Na_2O_2 also reacts with water to produce NaOH and H_2O_2 , which means that any $\text{Au}(\text{OH})_3$ formed will dissolve as soluble $[\text{Au}(\text{OH})_4]^-$, eqn (3). The amount of Na_2O_2 remaining in the PF cake can be qualitatively estimated from the degree of effervescence observed upon mixing the fusion cake with water, as H_2O_2 is unstable in alkaline solution, rapidly breaking down to H_2O and O_2 . It is not widely recognised that $\text{Au}(\text{III})$ may be reduced in alkaline digest solutions to colloidal Au^{31} (eqn (4)) leading to analytical losses of Au.²¹



Accordingly, the leaching of Au from the PF cake was investigated by treating ~ 0.4 mg specimens of flattened Au wire with 50:50 w/w $\text{Na}_2\text{O}_2 + \text{NaOH}$. Vigorous effervescence was observed when the resulting PF cakes were treated with water, producing solutions ~ 1 M in NaOH containing purple or greenish suspensions. This is consistent with the formation of $\text{Au}(\text{OH})_3(\text{s})$, which at $[\text{NaOH}] > 0.4$ M changes to sparingly soluble ‘greenish’ Na_2HAuO_3 .³² Such suspensions mostly dissolved within minutes but partial reduction of $\text{Au}(\text{III})$ to colloidal Au was indicated by the development of a brown turbidity that persisted even after acidification with HCl . Analysis of the acidified solution indicated losses of about 10% of the $\text{Au}(\text{III})$ content.

Replacing the water leachant with 2 M HCl produced a turbid brown suspension, which became a clear yellow solution within 2 hours. Subsequent testing after 48 hours confirmed quantitative Au recoveries, indicating that any colloidal Au formed during leaching of the fusion cake was dissolved by H₂O₂ acting as an oxidant in acidic solution, eqn (5).³³ All subsequent analytical work was performed using 2 M HCl to leach the fusion cakes.



3.3 Optimisation and application to CRMs

The general applicability of the PF/acid leach protocol was tested on a wide variety of finely divided ($\leq 45 \mu\text{m}$) CRMs, covering a wide range of mineralogies and Au concentrations. Using 50:50 w/w Na₂O₂ + NaOH and a flux weight eight times that of the sample produced fluid melts at 640 °C for all CRMs. The strong effervescence observed during HCl leaching of the fusion cakes indicated the persistence of an appropriate excess of Na₂O₂ during digestion. All CRMs could be brought into solution with a dilution factor, DF, of 50 (calculated as the final overall mass of the leach solution divided by the mass of the original sample) when leaching with 2 M HCl. However, most of these solutions gelled after several hours, presumably due to polymerisation of silicic acid.³⁴ Using a DF of 200 prevented gelling and gave solutions $\sim 1 \text{ M}$ in NaCl, $\sim 1 \text{ M}$ in HCl and $\leq 0.17 \text{ M}$ in Si(IV) that remained clear for several months.

The large DF required to ensure suitable solutions for analysis by GFAAS or ICP-MS necessitates a concentration/separation step to achieve a sufficiently low LOD and to minimise interferences from dissolved salts. Solvent extraction (SX) was considered to be the best approach for this purpose with methylisobutylketone (MIBK),^{26,27} dimethylisobutyl ketone (DIBK),^{11,26} and ethyl acetate (EtAc)^{35,36} the most likely options. All of these solvents readily extract Au from HCl solutions as HAuCl₄⁰, and are relatively benign from an OSH standpoint. Shaking each solvent with a solution of 2 M HCl + 1.5 M NaCl + 0.17 M Si(IV) showed that EtAc did not form emulsions, while those formed with MIBK could be easily broken by centrifugation. DIBK formed emulsions that were not readily broken. Because MIBK has a higher distribution coefficient (10^3 cf. 10^1 for EtAc), greater chemical stability towards HCl, lower aqueous solubility, and lower volatility this solvent was employed for all SX work.

Application of the optimised PF-AL-SX-GFAAS procedure to the CRMs gave Au concentrations within two standard deviations of the certified PbFA values (Table 3). It is particularly noteworthy that agreement between the current industry standard Pb-FA and the present protocol was achieved with CRMs covering the range of Au concentrations from barren silicate rock (OREAS 24b) to a copper-gold sulphide concentrate (OREAS 991).

Table 3: Comparison of results ($\mu\text{g-Au g-sample}^{-1}$) for Au in certified reference materials obtained by PbFA and by the present PF-AL-SX-GFAAS procedure.

Reference material	Certified PbFA			Found PF-AL-SX-GFAAS			
	Au ^a	SD ^a	RSD (%) ^b	Au	SD	RSD (%)	n ^c
OREAS 24b	0.002			<0.01	0.01		3
OREAS 45e	0.053	0.003	5	0.04	0.01		3
OREAS 901	0.363	0.018	5	0.31	0.02	6	6
OREAS 504b	1.61	0.04	14	1.6	0.1	6	6
OREAS 622	1.85	0.07	4	1.8	0.1	6	6
OREAS 210	5.49	0.14	8	5.7	0.5	9	6
OREAS 62e	9.13	0.41	2	9.4	0.1	1	3
OREAS 208	9.248	0.438	1	9.1	0.3	3	3
OREAS 991	47.04	0.22	1	46.6	0.4	1	3

^a Au was determined variously using AAS, ICP-AES, ICP-MS or gravimetry following cupellation. Au values and standard deviations are given as per the certificate of analysis.

^b The expected precision for analysis of 0.25 g samples calculated, according to the supplier's directions, from replicate neutron activation analyses supplied with the certificate of analysis.

^c Number of individual determinations using a sample weight of 0.25 g.

Clearly, the proposed PF-AL-SX-GFAAS protocol is as effective as PbFA in releasing Au from various mineral matrices and quantifying it. The procedure described here does not, of course, address the issue of the heterogenous nature of gold distribution in some materials but should be scalable for application to samples of >10 g. This is supported by the observation that RSDs were consistent with those expected from CRM inhomogeneity at the sample weights tested. The procedural LOD of 0.01 $\mu\text{g-Au g-sample}^{-1}$, calculated from fusion blanks and the effectively barren reference material OREAS 24b, demonstrates that the proposed method has adequate sensitivity for routine determination of Au at the $\mu\text{g g}^{-1}$ level in ores of widely varying mineralogy.

4. Conclusions

This study has developed a cleaner, safer, alkaline peroxide fusion protocol that produces complete dissolution of samples over a wide range of Au concentration and diverse mineralogies. As such it is a viable alternative to the current standard procedure of lead fire assay but significantly lowers waste disposal and OSH problems associated with that technique. Analysis of a range of certified reference materials gave excellent agreement with traditional PbFA determinations. The consumption of expensive Na_2O_2 was lowered substantially by the use of mixtures with NaOH without loss of efficacy or accuracy. Experiments with Au wire suggest that the proposed PF protocol will completely dissolve macroparticulate Au without additional sample treatment and so should lend itself to the analysis of the larger masses typically used in routine commercial Au determinations.

5. Acknowledgements

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6. Funding sources

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Highlights

3 to 5 bullet points (maximum 85 characters, including spaces, per bullet point)

Peroxide fusion GFAAS method suitable for routine commercial lab analysis developed

Results comparable to Pb fire assay over wide range of mineralogy and concentration

Potential for gold losses critically evaluated and found to be minimal

Addition of NaOH significantly reduces amount of Na₂O₂ required