under N_2 for 3.5 h in an oil bath maintained at 100 °C. The reaction mixture was poured onto an excess of ice, and after the ice had melted H_2O was added to bring the total volume to 1500 mL. The solution was extracted with CH_2Cl_2 (5 × 100 mL), and the combined organic layers were extracted with saturated $NaHCO_3$ until the extracts were colorless. The combined $NaHCO_3$ extracts were back-washed with CH_2Cl_2 (100 mL) and then acidified to pH 1 with 12 M HCl. The product was extracted into CH_2Cl_2 (5 × 100 mL), the organic layer was dried (Na_2SO_4) and filtered, and solvent was removed on a rotary evaporator followed by a vacuum pump to yield 6.3 g of 3-hydroxy-5-methylbenzocyclobutenedione in 63% overall yield from dienophile **5**: light yellow crystals; mp 191–193 °C (EtOAc); IR (CH_2Cl_2) 3540, 1789, 1755; ^1H NMR (60 MHz, acetone-d_6) δ 7.29 (s, 1 H), 6.95 (s, 1 H), 2.46 (s, 3 H). Anal. Calcd for $C_9H_6O_3$: C, 66.66; H, 3.73. Found: C, 66.56; H, 3.90.

Cycloaddition of 1-Methoxy-3-(trimethylsiloxy)-1,3-butadiene (13) to 1,4-Dichloro-3,3,4-trifluorocyclobutene (5). (A) Preparation of Enone 15. 1,4-Dichloro-3,3,4-trifluorocyclobutene (**5**; 3.08 g, 17.4 mmol) and Danishefsky's diene (**13**; 4.50 g, 26.1 mmol) were placed in a 3-oz Fischer–Porter pressure vessel equipped with a magnetic stirring bar, and the mixture was saturated with dry N_2 for 5 min. The tube was sealed, placed in an oil bath maintained at 120 °C, and stirred for 3.5 h. After cooling to 25 °C, the tube was opened, and the contents were transferred to a 100-mL round-bottomed flask with the aid of a small amount of MeOH. To this mixture was added 100 mL of 1:1 MeOH/1.2 N HCl, and the solution was stirred at room temperature for 2 h. The dark solution was poured into a separatory funnel, diluted with 200 mL of H_2O, and extracted with CH_2Cl_2 (3 × 75 mL), and the combined organic layers were dried (Na_2SO_4), filtered, and condensed on a rotary evaporator to yield 3.11 g of crude **14** as a dark oil. The compound is contaminated with diene decomposition products as well as some enone **15**, but the following spectroscopic absorptions of **14** are apparent: IR (CH_2Cl_2) 1720; ^1H NMR (60 MHz, $CDCl_3$) δ 4.72 (t, J = 5 Hz, ~1 H), 3.30 (s, ~3 H), 2.6–2.7 (m, ~4 H). Without purification, crude **14** (3.11 g) was placed in a 250-mL round-bottomed flask and dissoved in dry benzene (175 mL). After addition of *p*-toluenesulfonic acid (166 mg, 0.87 mmol), the mixture was refluxed under N_2 for 27 h, cooled to room temperature, transferred to a separatory funnel, washed with saturated $NaHCO_3$ (2 × 25 mL), dried (Na_2SO_4), filtered, and condensed on a rotary evaporator, and the residue was chromatographed on silica gel (3 cm × 0.75 m; 3:2 hexane/CH_2Cl_2) to yield enone **15**: 2.25 g (53% yield from dienophile **5**); white needles; mp 44–45 °C (sublimed); IR (CH_2Cl_2) 1695; ^1H NMR (60 MHz, $CDCl_3$) δ 6.63 (d, J = 10 Hz, 1 H with smaller splittings), 6.15 (d, J = 10 Hz, 1 H with smaller splittings), 3.90–3.13 (m, 1 H), 2.71 (apparent d, J = 4 Hz, 2 H); mass spectrum, m/e (relative intensity) 244 (M^+), 246 (M^+ + 2, 66).

(B) Aromatization of Enone 15 to 2,2-Difluoro-4-hydroxybenzocyclobutenone (16). A solution of enone **15** (2.25 g, 9.2 mmol) in 60 mL of MeOH was cooled to 0 °C in a 250-mL round-bottomed flask and the solution was saturated with dry N_2 for 5 min. The flask was fitted with a pressure equalizing addition funnel and a nitrogen inlet tube and the addition funnel was charged with a solution freshly prepared from sodium (0.846 g, 36.8 mmol) and MeOH (30 mL). The NaOMe/MeOH solution was added dropwise with stirring over 30 min, and after the addition was complete, the reaction mixture was allowed to warm to room temperature and stirred an additional 3 h. An equal volume of 1.2 N HCl (90 mL) was added, and the mixture was refluxed for 3 h to hydrolyze the intermediate ketal. After cooling to room temperature, the reaction mixture was diluted with 200 mL of H_2O and the product extracted into CH_2Cl_2 (3 × 75 mL). The combined CH_2Cl_2 layers were dried (Na_2SO_4), filtered, and evaporated to dryness on a rotary evaporator followed by a vacuum pump to give **16**: 1.5 g (96%); white crystals; mp 163–164 °C (EtOAc/hexane); IR (CH_2Cl_2) 3570, 1795, 1770; ^1H NMR (60 MHz, acetone-d_6) δ 9.8 (br s, 1 H), 7.70–7.16 (m, 3 H); mass spectrum, m/e 170 (M^+).

(C) Hydrolysis of 16 to 4-Hydroxybenzocyclobutenedione (17). The sample of **16** prepared above (1.50 g, 8.82 mmol) was placed in a 250-mL round-bottomed flask with 1:1 concentrated H_2SO_4/HOAc (90 mL) and stirred at 90 °C under N_2 for 3 h. The reaction mixture was poured onto an excess of ice and diluted with H_2O to a total volume of 350 mL. The solution was extracted with Et_2O (3 × 100 mL), and the combined organic layers were dried (Na_2SO_4), filtered, and condensed to a crude solid on a rotary evaporator. This material was chromatographed on a silica gel column (3 cm × 1 m; Et_2O) to yield 4-hydroxybenzocyclobutenedione: 1.10 g (84% yield, 43% overall from dienophile **5**); light yellow crystals; mp 174.5–175 °C (EtOAc) (lit.[7] mp 167–170 °C); IR ($CHCl_3$) 3600–3000 (br), 3575, 1808 (sh), 1790, 1769, 1755 (sh); ^1H NMR (60 MHz, CD_3CN) δ 7.81 (d, J = 8 Hz, 1 H), 7.36–7.06 (m, 2 H), 6.23 (br s, 1 H); mass spectrum, m/e 148 (M^+).

Acknowledgment is made to the National Cancer Institute, DHEW (Grant No. CA 26374), for support of this work.

Registry No. 1, 6383-11-5; **3**, 3469-06-5; **4**, 82431-14-9; **5**, 2927-72-2; **6a**, 63383-46-0; **6b**, 73912-36-4; **7a** (isomer 1), 82431-15-0; **7a** (isomer 2), 82468-19-7; **7b** (isomer 1), 82431-19-4; **7b** (isomer 2), 82468-20-0; **8a**, 82431-16-1; **8b**, 82444-39-1; **9b**, 82431-20-7; **10a**, 82431-17-2; **10b**, 82431-21-8; **11a**, 82431-18-3; **11b**, 82431-22-9; **12a**, 62416-21-1; **12b**, 82431-23-0; **13**, 59414-23-2; **15**, 82431-24-1; **16**, 82431-25-2; **17**, 75833-48-6; anthranilic acid, 118-92-3; 2-carboxybenzenediazonium chloride, 4661-46-5; 1,1-dichloroethylene, 75-35-4; 1,1-dichlorobenzocyclobutene, 68913-13-3; 3-methyl-2-butenal, 107-86-8; 2-methyl-3-buten-2-ol, 115-18-4.

Deperoxidation of Ethers. A Novel Application of Self-Indicating Molecular Sieves

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Received January 18, 1982

The removal of peroxides from contaminated ethers by treatment with self-indicating molecular sieves (IMS) is proposed as a safe and facile method of ether purification. Quantitative analysis of peroxide content before and after treatment with IMS show that ethers such as THF, diethyl ether, and diisopropyl ether can be readily decontaminated by an ambient-temperature or reflux process. The deperoxidation process is enhanced under nitrogen and has been safely carried out on a bulk scale and with initial peroxide contents as high as 0.5 M. IMS, in common with most other chemical reducing agents used for ether deperoxidation, are, however, ineffective for the decomposition of unreactive species such as dialkyl peroxides.

Aliphatic ethers, with their characteristic solvation abilities, excel as inert reaction media in numerous synthetic procedures. However, in practice this usefulness is often tempered by an unfortunate proclivity to facile air oxidation at ambient temperatures which leads to peroxide formation.[1] The presence of peroxides is not only po-

0022-3263/82/1947-3821$01.25/0

Table I. Deperoxidation of Tetrahydrofuran with Indicating Molecular Sieves (IMS) at Ambient Temperatures

run	scale, mL	sieve loading, % w/v	peroxide content,[c] mmol/L						
			initial	1 day	2 days	3 days	4 days	7 days	90 days
1	50	10	7.8		0.9		0.1	N.D.	
2[a]	50	10	7.8		3.3		0.5	0.26	
3	2400	5	1.1		0.22	0.10	0.06	0.014	*d*
4	100	5	1.1		0.07	0.007	0.018	0.012	*d*
5	50	5	22	7.8		5.5			
6[b]	50	5	22	7.1		4.9			
7	50	5	124		2.8				
8	50	10	176		14				
9	750	5	88	20	6		3		*d*
10	100	10	96	4					

[a] Performed under an atmosphere of air. [b] IMS activated at 300 °C in air oven. [c] As determined by the spectrophotometric method. [d] Not determined.

tentially hazardous,[1,2] but also frequently undesirable for chemical reasons, and although possible hazards may be avoided by the routine disposal of *time limit expired*[3] or *proven peroxide contaminated*[4] ethers, the need for rigorously purified solvents as well as economic and logistic[7] constraints may frequently necessitate peroxide removal. To this end a bewildering array[1,2a] of safe (and not so safe![8]) physical and chemical methods have been proposed.

Column chromatographic purification whereby peroxides are removed by adsorption on substrates such as alumina,[10] ion-exchange resins[11] or 13X molecular sieves[12] have variously been reported, and the use of activated alumina in particular has been widely endorsed.[2c,13] However, such methods, though effective and generally applicable, are disadvantaged by the necessity of using substantial amounts[14] of nonregenerable and relatively expensive adsorbents and the need for the subsequent safe disposal of the contaminated residue upon which the peroxides are adsorbed chemically unchanged.[10]

Chemical methods encompass a wide spectrum of reagents[1,2a] which effect reductive decomposition of peroxides. These include classical redox systems such as sulfites, bisulfites, amines, metal salts (e.g., $FeSO_4$, CuCl, $Ce(OH)_3$, $SnCl_2$), traditional reducing couples (e.g., Sn/HCl, Na/EtOH), and the more recently used complex metal hydrides ($LiAlH_4$, $NaBH_4$). Treatment with *aqueous*[15] ferrous sulfate,[2a,c,13a,d,16] or sodium sulfite/bisulfite[13a,d,16] has received approbation as a safe and effective method of deperoxidation, but their use is restricted to water-immiscible ethers,[18] and in any case such procedures necessitate an additional desiccation step. For water-miscible ethers such as dioxane, diglyme, and THF, reflux over solid cuprous[6b] chloride, stannous chloride,[13a,d] or $LiAlH_4$[6a] has been advocated. The use of these procedures, however, appears to be distinctly hazardous for heavily peroxidized ethers, and preliminary small-scale purifications are normally advised.[6a] Interestingly, an earlier method[19] employing solid cerous hydroxide, which is apparently effective and widely applicable, does not appear to be generally used probably because of the necessity for preparation of fresh reagent and the inaptitude of the methodology for application to bulk purification.

In the context of these various shortcomings, we propose the use of indicating molecular sieves (IMS) as an effective, safe, and readily available reagent which is generally applicable to the problem of peroxide removal from ethers.

Results and Discussion

Deperoxidation of Tetrahydrofuran (THF). The deperoxidation of THF was most thoroughly studied since of the common ethers discussed below this solvent has the highest proclivity toward peroxide formation.[20] Table I

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(1) See, for example, the brief review by N. V. Steere in "The Chemistry of the Ether Link", S. Patai, Ed., Interscience, London, 1967.

(2) See for example: (a) A. G. Davies, *J. R. Inst. Chem.*, 80, 386 (1956); (b) "Guide for Safety in the Chemical Laboratory", 2nd Ed., Van Nostrand-Reinhold, New York, 1972, p 302; (c) "Safety in Academic Chemistry Laboratories", 3rd ed., American Chemical Society, Washington, DC, 1979; (d) L. Bretherick, "Handbook of Reactive Chemical Hazards", 2nd ed., Butterworths, London, 1979.

(3) It has been proposed[6] that unused ethers in opened containers should be disposed of within 1 week of opening (uninhibited grades) or 3 or 6 months (inhibited grades). Elsewhere[2c] a blanket time limit of 1 month has been suggested for ethyl or isopropyl ethers. In one laboratory[6a] it is standard practice to use only THF from unopened bottles and to discard the remainder within 2–3 days.

(4) It has been recommended that diethyl ether[5] containing more than 0.005% peroxide (yellow color with KI test) and THF containing "larger than trace amounts of peroxide"[6] should be discarded.

(5) N. V. Steere, *J. Chem. Educ.*, 41, A575 (1964).

(6) H. E. Baumgarten, "Organic Syntheses", Collect. Vol. V, Wiley, New York, 1973: (a) p 796, (b) p 695.

(7) For laboratories situated outside the main industrial centers, chemical delivery times exceeding 6 months are not uncommon. As such ethers from unopened bottles are frequently significantly contaminated on receipt.

(8) Potassium hydroxide, which in its finely powdered form is a powerful desiccant,[9] has been advocated for peroxide removal. However, serious explosions may occur when treating impure THF with solid or concentrated aqueous potassium hydroxide.[6] Similarly, $LiAlH_4$, a less efficient drying agent,[9] has proved distinctly hazardous in the purification of ethers.[2d]

(9) D. R. Burfield, K. H. Lee, and R. H. Smithers, *J. Org. Chem.*, 42, 3060 (1977).

(10) W. Dasler and C. D. Bauer, *Ind. Eng. Chem. Anal. Ed.*, 18, 52 (1946).

(11) R. N. Reinstein, *J. Org. Chem.*, 24, 1172 (1959).

(12) N. Rabjohn, "Organic Syntheses", Collect. Vol. IV, Wiley, New York, 1963, p 475.

(13) (a) A. I. Vogel, "A Text-Book of Practical Organic Chemistry", 4th ed., Longmans, London, 1978; (b) "Purification of Solvents by Adsorbents Woelm", Woelm Pharma, Eschwege, West Germany; (c) "Drying in the Laboratory", E. Merck, Darmstadt, West Germany; (d) D. D. Perrin, W. L. F. Armarego, and D. R. Perrin, "Purification of Laboratory Chemicals", Pergamon, Oxford, 1980.

(14) The adsorbent loading required for effective deperoxidation is dependent on the grade of initial alumina and ether type, as well as on the initial water and peroxide content. However, for *dry* THF or dioxane of moderate peroxide content basic alumina of activity I will only purify less than an equal weight of ether.[13b,c]

(15) As opposed to cuprous and stannous chlorides, *solid* ferrous sulfate is apparently ineffective[16] in peroxide removal, and an explosion has been reported[17] on distilling THF from the solid salt. The effectiveness of solid inorganic reagents may perhaps be related to their solubilities in ethers since both cuprous and stannous chlorides are appreciably more soluble than the ferrous salt.

(16) A. C. Hamstead, *Ind. Eng. Chem.*, 56 (6), 37 (1964).

(17) J. Schurz and H. Stubchen, *Angew. Chem.*, 68, 182 (1956).

(18) The use of aqueous ferrous sulfate for deperoxidation of THF,[13a,d] is surely impractical as this ether is completely miscible with water.

(19) J. B. Ramsey and F. T. Aldridge, *J. Am. Chem. Soc.*, 77, 2561 (1955).

(20) This conclusion has been drawn from the analysis of some 30 samples of stored ethers of various types and from monitoring the rate of peroxide formation in purified ether samples exposed to the atmosphere.

Table II. Deperoxidation of Various Ethers with IMS

run[a]	ether	sieve loading, % w/v	peroxide content,[c] mmol/L							conditions[d]
			initial	3 h	1 day	2 days	3 days	7 days	90 days	
1	dioxane	10	1.7		0.22			0.44		A
2		10	5.5					4.0	0.6	A
3		5	10.3	0.61						R
4		5[b]	10.3	0.44						R
5	diethyl ether	5	12.8		0.05	e				A
6		5	12.8	0.24						R
7	diisopropyl ether	10[c]	53			0.3				A
8		10	109				1.1			A
9	trimethylene glycol dimethyl ether	10	29			5.5				A
10		5	27	<0.1						H
11	dibenzyl ether	5	37	<0.15						H

[a] Scale = 100 mL except as indicated. [b] Scale = 1500 mL. [c] Scale = 50 mL. [c] As measured by spectrophotometric method; [d] A = ambient temperature, R = at reflux, and H = at ca. 110 °C. [e] Not determined.

summarizes some representative results for the addition of IMS to peroxide-contaminated THF obtained from a variety of sources. In every case addition of IMS leads to a reduction in the peroxide content which decreases to a negligible level over extended periods of time (runs 1, 3, 4, 9). The efficiency of deperoxidation is enhanced under nitrogen. (cf. runs 1, 2) but is somewhat reduced by increased scale (cf. runs 3, 4).

Activation of the supplied IMS by heat treatment at 300 °C only marginally increases the efficiency of deperoxidation (runs 5, 6). However, deactivation of the IMS, by exposure to the atmosphere until the blue indicator decolorizes, renders this material *completely impotent* for peroxide removal.[21]

Deperoxidation of THF under reflux proceeds far more rapidly than by static deperoxidation at ambient temperatures. Thus peroxide contents as high as 500 mmol/L were dramatically reduced (95% peroxide removal) within 4 h of refluxing over 5% w/v IMS.

Deperoxidation of Various Ethers. The effects of ambient temperature and reflux treatment of various ethers with IMS are summarized in Table II. Generally at ambient temperatures peroxide removal from dioxane is relatively inefficient and considerably slower than for THF. On the other hand, under these conditions, deperoxidation of diethyl ether and diisopropyl ether is facile, proceeding more readily than with THF.

At reflux temperatures the deperoxidations of the various ethers are considerably accelerated, the effect being most noticeable for the higher boiling ethers. It is conceivable that this may in part be due to an uncatalyzed thermal decomposition. At the same time the effects of scale become less apparent (cf. runs 3, 4), and 1.5 L of dioxane is decontaminated as rapidly as 0.1 L. Refluxing diethyl ether shows little advantage over the comparable static purification, which in any case is already very efficient.

Nature of Peroxide Removal. Earlier studies[12] have shown that ethers may be deperoxidized by selective adsorption with 13X (10 Å) molecular sieves. Purification of THF with nonindicating sieves of varying pore sizes (Table III) confirms the possibility of preferential peroxide adsorption for 10A and to a lesser extent for 5A sieves. However, sieves of 4-Å pore size, i.e., equivalent in pore structure to the IMS, are completely ineffective. The effectiveness of IMS for deperoxidation must therefore be attributed to some alternative mechanism, and it seems not unreasonable to suppose that peroxides are decomposed by interaction with the impregnated indicator, which is probably a cobalt salt, through redox reactions such as:

Table III. Effect of Sieve Type on THF Deperoxidation

run[a]	sieve type	peroxide content,[b] mmol/L	
		15 days	30 days
1	no added sieve	8.7	14.8
2	4A	7.6	19.6
3	5A	2.9	3.1
4	10A	0.14	0.16
5	4A (with indicator)	0.26	

[a] Scale 50 mL; sieve loading 10% w/v; at ambient temperature under air. [b] As determined by the spectrophotometric method. The initial content was 7.8 mmol/L in all cases.

Table IV. Dialkyl Peroxide Content of Deperoxidized Ethers

sample	peroxide content, mmol/L		
		after treatment[c]	
	initial[a]	ROOH[a]	total peroxide[b]
THF	124	0.95	2.4
	96	0.60	2.8
diisopropyl ether	109	1.1	3.3
dioxane	52	e	17
diethyl ether	~1	0.2	1.1

[a] Analysis by spectrophotometric method. [b] Analysis by acid reflux. [c] Treated with 5% w/v IMS at ambient temperature. [e] Not determined.

$$ROOH + Co^{2+} \rightarrow RO\cdot + {}^{-}OH + Co^{3+}$$

The implication of this conclusion is that in contrast to the residues from normal chromatographic purifications, the IMS should be peroxide free and thus capable of hazard-free disposal or reactivation.

Formation, Detection, and Removal of Dialkyl Peroxides. Most methods employed for testing ether purity, including the standard acidified KI test,[22] and the

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(21) IMS are similarly ineffective for the treatment of grossly wet (>1% w/v water) ether samples, wherein water uptake by the molecular sieves leads to deactivation. Such samples require either predrying or addition of higher loadings (>5% w/v) of IMS to prevent saturation and deactivation of the indicator.

(22) In this test peroxides are deemed present if a faint yellow color develops on shaking 5 mL of the ether with an equal volume of 2% w/v KI solution in the presence of a few drops of dilute hydrochloric acid. (The detection limit of this test in our hands corresponds to a value of about 0.2 mmol/L as measured by the spectrophotometric method.

(23) R. D. Mair and A. J. Graupner, *Anal. Chem.* 36, 194 (1964).

spectrophotometric method used in this study are ineffective for the detection of dialkyl peroxides. The significance of this observation is that all the methods reported in the literature to date for the removal of peroxides have in fact been proven only for hydroperoxides.

For evaluation of the relative importance of dialkyl peroxide formation, several badly contaminated ether samples were deperoxidized with IMS and the decontaminated ethers analyzed for both *active* peroxide (hydroperoxide) and dialkyl peroxide (Table IV). In every case, except dioxane, the total peroxide content is very much reduced by treatment with IMS. However, the acid-reflux analysis indicates the presence of small but detectable amounts of dialkyl peroxide in these samples. In the case of dioxane there is a very large concentration (>30% of original content) which is not analyzed by the spectrophotometric method and is not decomposed by IMS during the period of treatment. It would appear, therefore, that oxidation of dioxane leads to the formation of much higher concentrations of dialkyl peroxides than other ethers or to hydroperoxides which are much less reactive.

In summary, it appears that predominant contaminants of ethers such as THF and diisopropyl and diethyl ethers are *active* peroxides which can be readily removed by treatment with IMS. Deperoxidation of dioxane is very much less efficient. Almost certainly, the inapplicability of IMS to the problem of dialkyl peroxides is also a limitation with other commonly used redox purification systems such as those based on CuCl, $SnCl_4$, $FeSO_4$, etc.

Applicability of IMS to Ether Deperoxidation. The prime criteria for deperoxidation methods are effectiveness and safety. Since most purifications are accompanied by distillation, which technique already allows a means of peroxide removal, the key aspect is safety. In this connection, IMS have proved effective in the deperoxidation of ethers with peroxide concentrations as high as 0.5 M without hazard. Compared to other methods IMS provide a relatively slow rate of peroxide decomposition, and this feature is almost certainly an important aspect of safe deperoxidation.

Since it has already been shown that molecular sieves are particularly effective in removing water[24] and other polar impurities[25] from ethers, as applied to diethyl ether for example, the use of IMS provides a safe and effective method of concomitant deperoxidation, deethanolization, and drying of the solvent. In the case of dioxane or other ethers where the presence of significant quantities of alkyl peroxide are suspected, IMS may be used for pretreatment before more rigorous deperoxidation is executed.

Experimental Section

Materials. Indicating activated Type 4A molecular sieve,[26] (4–8 mesh) were kindly supplied by J. T. Baker Chemical Co. and were used as received. Ethers were typical reagent grade solvents. High peroxide levels were induced by aging purified samples in the presence of air over a period of days or weeks at ambient temperatures.

Deperoxidation Method. (i) Static Deperoxidation. Ethers, in their original containers, were first deoxygenated by bubbling with oxygen-free nitrogen for about 5 min in a fume cupboard. Subsequently, 5% w/v of indicating molecular sieves was added and the ether tightly capped and set aside.

(ii) Reflux Deperoxidation. Ethers were charged into a distillation flask and deoxygenated with a slow stream of nitrogen. Subsequently, 5% w/v of indicating molecular sieves was added and the ether brought slowly to reflux under nitrogen.

Peroxide Analysis. (i) Spectrophotometric Analysis. Hydroperoxides were analyzed by a modified spectrophotometric method based on the procedure of Wagner et al.[27] This ferrous ion oxidation method permits quantitative analysis of hydroperoxides (confirmed for *tert*-butyl hydroperoxide) but is completely insensitive to dialkyl peroxides such as di-*tert*-butyl peroxide.

(ii) Total Peroxide Analysis. An acid-reflux method proposed by Mair and Graupner[23] was used and was found to provide a quantitative assay for dialkyl peroxides such as di-*tert*-butyl peroxide.

Acknowledgment. I acknowledge the assistance of Mr. Lee Meng Lay in conducting the peroxide analyses.

Registry No. Tetrahydrofuran, 109-99-9; dioxane, 123-91-1; diethyl ether, 60-29-7; diisopropyl ether, 108-20-3; trimethylene glycol dimethyl ether, 17081-21-9; dibenzyl ether, 103-50-4.

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[24] D. R. Burfield, G. H. Gan, and R. H. Smithers, *J. Appl. Chem. Biotechnol.*, **28**, 23 (1978).

[25] D. R. Burfield and R. H. Smithers, *Chem. Ind.* (*London*), 240 (1980).

[26] Indicating molecular sieves (Type 4A) as supplied by Merck and Sigma were found to be equally effective. The product of the latter company is, however, only provided as a mixture (approximately 10% w/w) with nonindicating molecular sieves. Generally smaller bead sizes are more efficient for deperoxidation due to increased surface area.

[27] C. D. Wagner, H. L. Clever, and E. D. Peters, *Anal. Chem.*, **19**, 980 (1947).